

CryoProbe System

Installation

Version 1

BRUKER

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Safety

1

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

Terms and symbols

1.1

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



Figure 1.1. *Hot surface!*

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



Figure 1.2. *High voltage!*

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



Figure 1.3. *Dangerous device!*

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



Figure 1.4. *Cold surface!*

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



Figure 1.5. *Wear protective gloves!*

Put on protective gloves before handling the labelled item.



Figure 1.6. *Wear protective goggles!*

Put on protective goggles before handling the labelled item.



Figure 1.7. *Strong attraction by magnet!*

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

Disclaimer

1.2

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

Emergency

1.3

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

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

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

See the *CryoProbe System User Manual* for details.

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

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

If in doubt: Put on goggles and protective gloves!

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

Inherent safety

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

CE certification

CryoProbe, CryoCooling Unit and He Compressor are CE certified.

Technically qualified personnel only

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

No user-serviceable parts inside

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

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

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

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

Pressurized cold helium gas cycle

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

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

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

Pressurized helium gas supply

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

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

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

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

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

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

Overpressure release noise

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

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

Electrical safety

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

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

No hazardous substances

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

Lifting the CryoProbe

WARNING: Two persons 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 persons 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.

Safety of CryoProbe equipment

1.5

CAUTION:

- Do not bend the CryoProbe.

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

- Do not open the CryoProbe.

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

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

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.

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

How to use this manual

2.1

This *CryoProbe System Installation Manual* guides you through the initial setup and test of a CryoProbe System. Several procedures are the same as in daily use, so they are described in the *CryoProbe System User Manual*. The two manuals are complementary and must both be available for an installation.

Use the appropriate **"Check lists" on page 75** to keep track of your actions.

If you have a specific question, use

- ***"Contents"***,
- ***"Index"*** or
- ***"Frequently asked questions"***

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 79**.

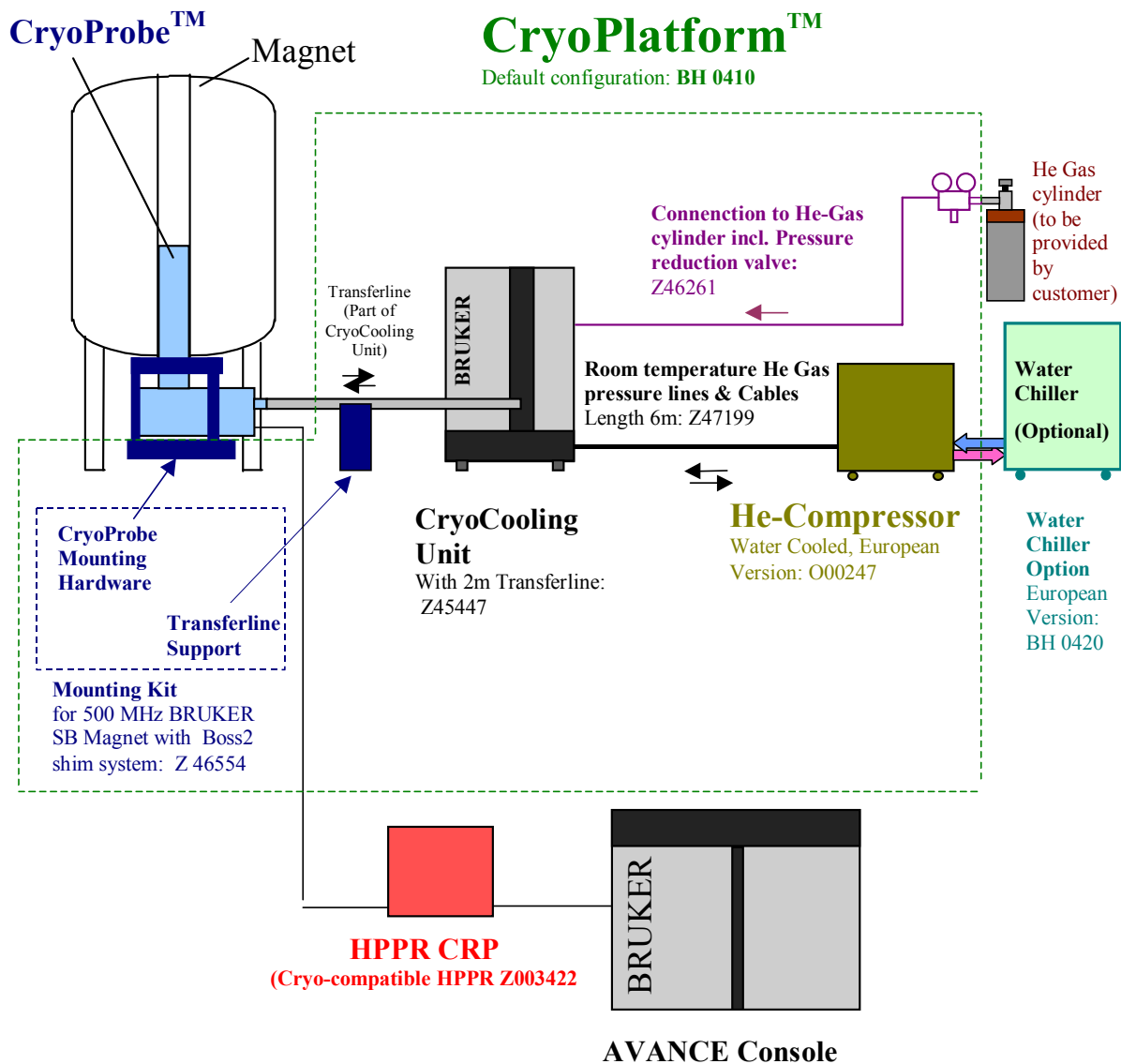
CryoProbe System overview

2.2

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

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

Figure 2.1. The CryoProbe™ System



Conventions

2.3

<i>SMALL CAPS ITALIC</i>	setting of a hardware switch or button
Courier small	contents of a file
<i>Courier small italic</i>	system response
Courier	file or directory name
Courier bold	Unix™ or Windows™ NT keyboard command

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

Before an installation date with BRUKER is fixed, the customer needs to read the following remarks and work through the **"Site preparation checks" on page 75** for the NMR laboratory.

The checklist **"Preparations for the installation visit" on page 75** is intended for the BRUKER engineer.

General

All necessary preparations for the CryoProbe System must have been completed before BRUKER engineers arrive at the customer site and perform the installation. BRUKER personnel will connect all units of a the CryoProbe System as far as required for test and operation but they will not work on the laboratory infrastructure (e.g. mount cable channels, fix tubes to the wall, drill holes or the like) to make the installation 'look nice'.

NOTE: The spectrometer cannot be used for NMR experiments (with e.g. a conventional setup) during installation and test of the CryoProbe System.

Other laboratory equipment

Other devices in the laboratory may depend on the same supplies as the CryoProbe System, e.g. pneumatic gas and electricity. It must be checked if these devices can continue their operation while the CryoProbe System is hooked up to the shared supplies.

Reference data

Get a set of reference spectra with a conventional probe to estimate spectrometer performance and external disturbances. The spectra should be fairly new. Check e.g. lineshape, sensitivity, water suppression, lock stability spikes and vibrations, t_1 -noise etc.

NOTE: Sensitivity is the major issue for the CryoProbe and it is influenced by numerous external factors. The CryoProbe cannot reach its specs if any other spectrometer component fails to do so!

The CryoProbe System is shipped in three steps: first a *Site Preparation Set* containing the He Compressor and some tubes which must be installed by the customer in the NMR lab to provide the infrastructure for the CryoProbe System. After the site preparation work has been completed, CryoCooling Unit and HPPR CRP are sent off and installed by a BRUKER engineer. Finally, the CryoProbe is deliv-

ered and installed in a third step. If circumstances permit, the second and third step are done at together.

Crate dimensions

All dimensions in [mm], width × length × height.

- 1.a wooden crate 770 × 600 × 930, 150 kg water-cooled He Compressor,
or
 - 1.b wooden crate 820 × 700 × 1130, 170 kg: air-cooled He Compressor,
 - 2. wooden crate 1210 × 1380 × 1520, 620 kg: CryoCooling Unit,
 - 3. cardboard box 350 × 440 × 510, 27 kg: HPPR CRP,
 - 4. cardboard box 1200 × 800 × 500,
pallet 1200 × 800 × 140,
flightcase 1010 × 510 × 340, 30 kg
i.e. in total 1200 × 800 × 640, 40 kg: CryoProbe,
- + some smaller items in cardboard boxes.

The crates are not returned to BRUKER but preferably kept at the customers site.

CAUTION: All components of a CryoProbe system are fragile! The CryoCooling Unit must be shipped in upright position. There are shock watches and tilt indicators on its shipping crate.

Transport pathways

Check transport pathways and transportation means before sale. Do size or weight of the CryoProbe System exceed the capabilities of elevators, floors etc.? Usually, the same transport pathway as used for the magnet delivery will be fine (see Avance Site Planning 200-700, § 1.5). A pallet truck suffices as transport aid.

If the transport pathway in the destination building is too narrow for the CryoCooling Unit shipping case, this unit might be unpacked and carried on a pallet truck to its final site. Then, all passages must be only at least 100 cm wide. Note that the pallet truck does not go straight under the CryoCooling Unit but only at about a 45° angle.

WARNING: Avoid magnetic transport aids when moving devices inside the magnet's 0.5 mT stray field. Even heavy equipment like a pallet truck may be attracted and cause fatal damage.

Environmental conditions for transport and storage

The allowed ranges of temperature, humidity, atmospheric pressure, dust, and maximum accelerations are the same as for AVANCE spectrometers.

Check the site preparations

3.3

Have all necessary preparations been done in the lab? Are all supplies available? Use the ***"Site preparation checks" on page 75.***

Ensure safety

Read the instructions for "**Safety**" on page 7 before starting the installation.

Check instrument cases for damage

Inspect the cases, shock watches, and tilt indicators.

In case of damage: Do not unpack! You need to do certain things now for legal reasons. Call your BRUKER service office and ask for advice:

- Should you accept the goods or refuse them? (Note: Usually, BRUKER is the 'customer' of this transport, but not BRUKER's 'NMR customer' who owns the lab.)
- Do you need to write a statement?
- Is the visit of an insurance expert necessary?
- Should you unpack and install?

Record actual NMR spectrometer status

Mark the incoming gas pressure on the gauge at the rear of the spectrometer cabinet with a waterproof pen or write down the reading with a precision of 0.1 bar. If there is a pressure gauge on the magnet suspension, mark or record that value, too.

Is a NMR performance reference data set available (sensitivity, lineshape, water suppression, etc.)? Are any spectral artifacts visible like spikes or noise?

IMPORTANT: Before installing the CryoProbe System, make sure there are no performance problems with the conventional parts of the spectrometer. The CryoProbe cannot reach its specifications if any other spectrometer component fails to do so!

Use the shipping and packing lists to check if all parts arrived and if they are complete. In case of obvious damage, proceed according to the recommendations given in "**Check instrument cases for damage**" on page 19. Keep all packing material and transport certificates until the CryoProbe System runs satisfactorily. The CryoProbe flight case is ideal for storing the CryoProbe when not in use.

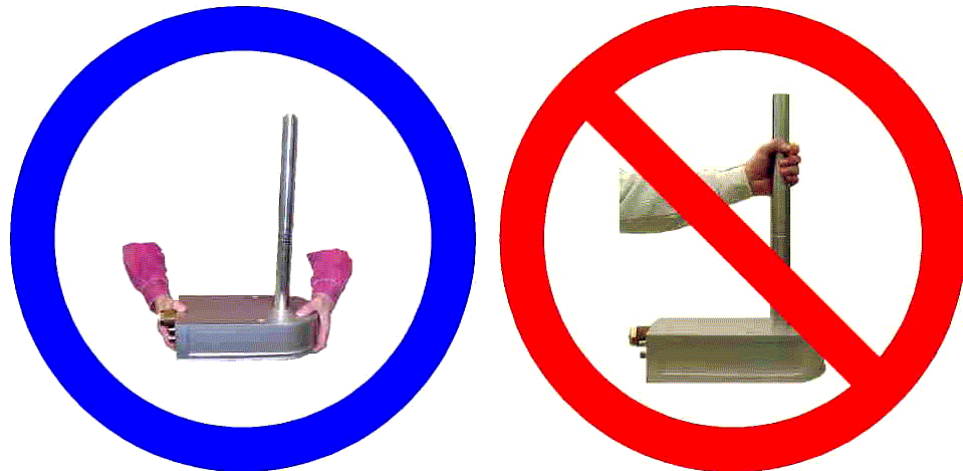
CryoProbe

Keep the CryoProbe in its flightcase until you can mount it into the magnet. Leave the protective caps on sample cavity, CryoCoupler, and RF sockets until you make the connections.

In the upper right corner of the CryoProbe front, there is a little hole with an indicator screw inside. This screw must not stick out but be flush with the front plate. If the screw 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.

CAUTION: The CryoProbe is heavy (~12 kg) but fragile. Hold it only at its body, not at the tube. Never bend the tube!

Figure 3.1. CryoProbe handling



If only an additional CryoProbe is to be installed on a working CryoPlatform, continue with **"Handling" on page 27.**

CryoCooling Unit

Disassemble the crate and remove the transport fixtures. The CryoCooling Unit weights ~400 kg. It is most convenient to lift it off the pallet with a fork-lift truck. It can also be lifted with a hoist. There are four eyes (for hooks or ropes) on the inner frame of the CryoCooling Unit. To access them, remove the top panel of the CryoCooling Unit (see **"How to open the CryoCooling Unit" on page 67.**)

IMPORTANT: Take special care of the He Transferline when unpacking and moving the CryoCooling Unit. Avoid tight bending! If the radius becomes smaller than 0.7 m, i.e. if a 180° turn has less than 1.4 m diameter, its internal structure can be damaged.

On the back panel, there's a plug without cable connected to REMOTE SWITCH. Make sure this plug is in place. If the plug is missing, the mains of the whole CryoCooling Unit is inhibited.

He Compressor

Check the SUPPLY PRESSURE gauge on the He Compressor front. It should indicate about 0.1 bar (i.e. 0.01 MPa \approx 0.1 kgf/cm²). If the pressure gauge reads plain 0 bar, contact BRUKER: this is a major problem and a lengthy gas purifying cycle is required.

Water-cooled He Compressor (P/N O00247) only: Set the selector switch according to the available mains voltage and frequency. The switch is hidden behind the right side panel. See section 2.5 in the He Compressor operation manual which is delivered with the CryoPlatform.

NOTE: On the backside of the He Compressor, keep the protective cap on the **HELIUM GAS CHARGE**. This connector is never used.

WARNING: Avoid magnetic transport aids when moving the CryoCooling Unit to its position inside the magnet's 0.5 mT stray field. Even heavy equipment like a pallet truck may be attracted and cause fatal damage.

Table 3.1. Position the units

step	action
p.1	<p>Place all units in the laboratory as agreed on during site planning: CryoCooling Unit with He Transferline, Transferline Support, He Compressor, and optional water chiller & radiator.</p> <p>Install the He Compressor on a level surface. Two of its four casters should be locked.</p> <p>Provide extra space around the units as required for connections, ventilation, and service (details are given in the <i>CryoProbe System Site Planning Guide</i>).</p>
p.2	<p>NOTE: Orientation of the Transferline Support: the screw fixture of the vacuum tube must face the CryoCooling Unit.</p> <p>Connect the shorter vacuum tube from the Transferline Support to the vacuum port at the CryoCooling Unit front, right below the He Transferline outlet.</p> <p>NOTE: On the earliest CryoCooling Units, the vacuum port is not accessible from outside. Open the cabinet (see <u>"How to open the CryoCooling Unit" on page 67</u>) and connect the <i>longer</i> (!) vacuum tube to the port inside.</p>
p.3	<p>Lock the He Transferline to the Transferline Support with the four big screws.</p>
p.4	<p>Adjust the positions of CryoCooling Unit and Transferline Support such that the He Transferline will easily fit into the CryoProbe when bent by 100°-140° (the bending is needed because it reduces the transfer of vibrations to the magnet). Consider that the tip of the CryoCoupler will be located about 10 cm away from the magnet's symmetry axis when connected inside the CryoProbe.</p> <p>NOTE: The heavy inner frame of the CryoCooling Unit rests on three feet. Thus, it needs no balancing. The four feet in the corners are not intended to actually touch the floor. They just add security against toppling and need no adjustment.</p>
p.5	<p>Put the dark-grey basement cover to the CryoCooling Unit front bottom.</p>

BCU05 cooling test (optional)

3.7

If a gas cooler, e.g. a BCU05, shall be used with a VT hose between its outlet and the CryoProbe, its output temperature must be tested and adjusted. The goal is that the VT gas enters the CryoProbe no colder than the lowest permitted sample temperature. This temperature is specified on the CryoProbe's LIMITATIONS - WARNINGS sheet. The test can be run conveniently while the CryoProbe System is installed.

In the presence of an isolated VT gas port that is integrated with the Tuning Adapter, this BCU05 cooling test can be skipped.

Table 3.2. Adjust the BCU05 outlet temperature

step	action
b.1	Connect the spherical adapter of the CryoProbe VT hose to the BCU05 gas outlet but do not connect the VT hose to the CryoProbe.
b.2	Set the gas flow rate to the minimum value specified on the CryoProbe's LIMITATIONS - WARNINGS sheet (e.g. 600 L/h).
b.3	Switch ON the BCU05.
b.4	After about 1 h , the BCU05 will have reached its operating temperature and part of the CryoProbe VT hose will be covered with ice .
b.5	Shorten the VT hose from its 4 mm end such that its outlet is just not frozen or moist. NOTE: Keep at least 10 cm of the 4 mm tube! If necessary, continue to chop off at the 8 mm tube.
b.6	Repeat b.4 to b.5 until the 4 mm outlet of the VT hose is just not frozen .

Spectrometer modifications

3.8

Remove any sample and conventional probe from the magnet. Close the magnet bore temporarily to protect it against magnetic particles.

Access to the magnet bottom

3.8.1

A spatial channel of at least 195 × 578 (628) mm [width × height] is needed from the magnet front to insert a 500 (600) MHz CryoProbe (see *Site Planning Guide*).

NOTE: A QNP pneumatic unit prevents the installation of a CryoProbe and must be removed for the time of CryoProbe operation.

Special cranked bars are necessary only for BRUKER/Spectrospin 500 MHz and 600 MHz magnets if they rest on an anti-vibration stand with horizontal pillar braces. The cranks enlarge the vertical gap between the bars such that a CryoProbe can be introduced.

WARNING: Do not remove any pillar brace before the magnet stand was locked! Do not remove both braces at once because the magnet stand will be less stable then and a misalignment of the magnet stand can result!

Necessary parts:

- 1 cranked upper pillar brace (Z55762)
- 1 cranked lower pillar brace (500 MHz: Z55761, 600 MHz: Z55763)
- 4 M8×25 Allen key screws, non-magnetic (14460 each), for upper brace (Use the existing four Allen key screws for the lower pillar brace)
- 1 6 mm Allen key wrench, non-magnetic
- 9 M8×35 Allen key screws, non-magn. (28059 each), came with magnet

If the magnet stand height needs to be adjusted:

- 1 open end wrench 24 mm (500 MHz) or 30 mm (600 MHz) (e.g. Z56883)

Table 3.3. Mount the cranked magnet pillar braces (optional)

step	action
s.1	<p>Check the gap between the lower magnet pillar brace and the floor: it must be at least 95 mm.</p> <p>If necessary, adjust the height of the magnet's feet at their thread right above the floor. For this, remove the inside covers from the pillars. Lock the two nuts against each other with a non-magnetic open end wrench. Use the nuts to turn the threaded bar itself. See also the drawing <i>Mechanical Levelling of the Anti Vibration Stand</i> in the magnet manual.</p>
s.2	<p>Put 9 fixing screws for the magnet stand into place (delivered with the magnet, M8×35 Allen key, non-magnetic), three for each damper: insert them from the top and tighten them firmly. If the holes are not aligned you may move the anti-vibration column with some force. Do not manipulate the three large screws in each center.</p>
s.3	<p>Switch OFF the magnet's air suspension.</p>
s.4	<p>WARNING: Do not remove a brace if the magnet is still suspended or if the 9 fixing screws are not in place!</p> <p>Replace one of the two front braces while the other brace is still in place. The lower brace has the larger kink and will almost touch the floor. Orient the braces the same way as the original braces were.</p>
s.5	<p>Replace the second brace only after the first brace has been tightened.</p>
s.6	<p>Remove the 9 fixing screws from the magnet stand.</p>
s.7	<p>Do not switch <i>ON</i> the magnet's air suspension yet but wait until the CryoProbe is mounted.</p>

CAUTION: Check if a blue sample spinning stator is mounted inside the shim system but not a red one (blue stators were introduced shortly after delivery of the first AVANCE spectrometers started). Only blue stators are compatible with the CryoProbe, others may damage its top. A blue stator is backwards compatible with all conventional BRUKER probes including all old models.

The color of the stator can be easily seen from below the magnet when using a flash light. Look into the shim system bore while no probe is in the magnet.

If the shim system is not equipped with a blue spinning stator, it must be replaced as a whole.

NOTE: All previous shimfiles will become invalid. However, if the new shim system is mounted at exactly the same position as the old one, the accompanying change is not expected to be large, thus the old shimfiles are valuable start files for shimming.

Necessary parts:

- 1 BRUKER shim system with blue sample spinning stator
- 1 3 mm Allen key, non-magnetic, delivered with magnet
- 1 4 mm Allen key, non-magnetic, delivered with magnet

Table 3.4. Replace the shim system (optional)

step	action
g.1	Remove any sample or probe from the magnet.
g.2	Switch OFF the magnet's air suspension.
g.3	Mark the exact position of the shim system with a permanent marker or small scratches at the magnet bottom and on the shim upper part but not on the shim system (the red clamping ring at the magnet bore bottom will be removed permanently!).
g.4	Release the three single-slit screws that are accessible from the very top of the shim upper part. Do not untie the red clamping ring from the shim upper part at the top of the magnet because it determines the correct position of the shimcoils. CAUTION: Do not touch the Allen key screws in the magnet's flat top flange!

g.5	<p>Remove the red clamping ring from the magnet bottom and let the shim system slide out. If there is an optional ring next to it for compressed air to cool/heat the shim system, it must be removed also.</p> <p>CAUTION: Do not touch the Allen key screws in the magnet's flat bottom flange!</p> <p>NOTE: For a usual shim system exchange, one would leave the red rings in place, but in this case they will be replaced later anyhow in <u>"Attach the Mounting Hardware" on page 25</u>.</p>
g.6	Put the new shim system into the magnet.
g.7	Fasten the three single-slit screws at the sample lift top tightly.
g.8	Check if the position of the shim system still agrees with the marks.
g.9	Do not switch <i>ON</i> the magnet's air suspension now but wait until the CryoProbe is mounted. To fix the lower part of the shim system permanently proceed with <u>"Attach the Mounting Hardware" on page 25</u> .

Attach the Mounting Hardware**3.8.5**

The CryoProbe is too heavy for being fixed at the shim system bottom plate. It requires a special Mounting Hardware that ties it directly to the magnet.

Necessary parts:

- 1 CryoProbe Mounting Hardware (Z44865)

NOTE: For some Magnex magnets with a larger radius of the baseplate screws (40 mm instead of 38 mm) the parts Z49989 and Z49987 are needed additionally.

- 4 M5×12 Allen key screws, non-magnetic (P/N 11125 each)
- 1 3 mm Allen key, non-magnetic, delivered with magnet
- 1 4 mm Allen key, non-magnetic, delivered with magnet

If a ring for cooling/heating the shim system with compressed air is present around the shim system bottom or if no prepared threads are found in the magnet bottom flange, use

- 4 M5×20 Allen key screws, non-magnetic (11127 each) instead of M5×12

Table 3.5. *Attach the Mounting Hardware*

step	action
h.1	Switch OFF the magnet's air suspension.

h.2	<p>Check if the shim system and the shim upper part (i.e. the sample lift tube) are fixed to the magnet top and bottom with red clamps. The upper clamp must be present at all times and the 3 long vertical screws in the shim upper part top must be tightly fixed.</p> <p>Mark the exact position of the shim system at the magnet top with scratches or a permanent pen.</p>
h.3	<p>Remove the red shim system fixture clamp at the magnet bottom.</p> <p>NOTE: If an optional ring for cooling/heating the shim system with compressed air is mounted at the magnet bore bottom, it can be left in place but the four Allen key bolts (M5×12) that tie it to the magnet must be removed.</p>
h.4	<p>NOTE: In rare cases, it may be necessary to rotate the shim system permanently to resolve conflicts between Mounting Hardware, shim system, desired CryoProbe orientation, and magnet parts like drop-off plates. See the <i>CryoProbe System Site Planning Guide</i> for a 1:1 drawing of the Mounting Hardware interface plate (the interface plate is the part that touches the magnet).</p> <p>Before rotating the shim system, mark its old position clearly. Release the upper clamp just a little bit from the shim upper part. Turn the shim upper part gently into its new position and fix all screws tightly. Take care not to let it slide into the magnet!</p>
h.5	<p>Check if there are four empty threads in the magnet bore bottom flange at a diameter of 76 mm.</p>
h.6	<p>NOTE: Sometimes it may be necessary to rotate the shim system temporarily to access the four threads or screws through the holes in the shim system bottom plate.</p> <p>If the shim system needs to be rotated, mark its original position clearly. Remove the four screws from the upper clamp but keep the clamp tightly fixed to the shim upper part. Turn the shim upper part gently into its temporary position.</p>
h.7	<p>Take the CryoProbe support plate out of the Mounting Hardware.</p>
h.8	<p>Remove the clamp part that is integrated in the interface plate.</p>
h.9	<p>Fix the interface plate at two of the open threads at the magnet bore such that the clamp opening points in the same direction as the CryoProbe front will. Do not tie the screws firmly yet.</p> <p>NOTE: There are two cases in which four longer non-magnetic Allen key screws (M5×20) must be used instead of M5×12:</p> <ol style="list-style-type: none"> An optional ring for cooling/heating the shim system is present (see step h.3): mount the interface plate such that the cooling ring is sandwiched between magnet and Mounting Hardware. If screws had to be removed from the magnet bottom flange to provide four empty threads (see step h.5).

h.10	<p>Insert the clamp part, put the remaining two screws in place, and tie all six screws in a balanced fashion.</p> <p>IMPORTANT: Make sure that the shim system tube is not tilted inside the magnet bore. Center it properly to avoid shimming problems.</p> <p>If the shim system had been rotated temporarily in step h.6, release the six screws again just a little bit. Bring the shim system back to its original orientation and fix it tightly, then tie the six screws again.</p>
h.11	<p>Do not switch <i>ON</i> the magnet's air suspension now but wait until the CryoProbe is mounted.</p>

Handling

3.9

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

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

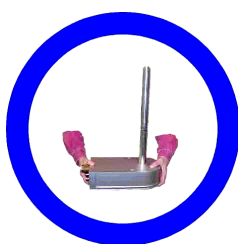


Figure 3.2. Carry a CryoProbe only at its body!



Figure 3.3. 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).

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

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

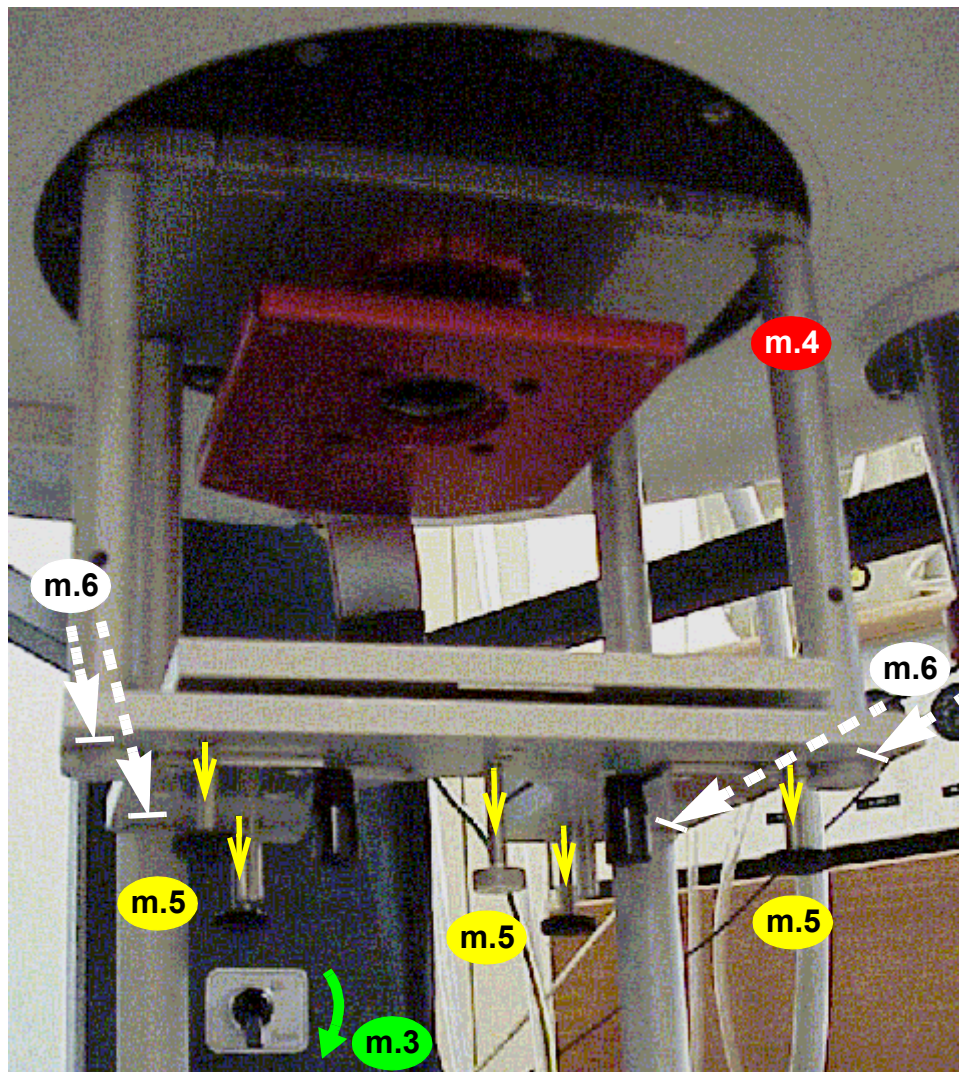
IMPORTANT: Read **"Handling" on page 27** if you're going to handle a CryoProbe for the first time.

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

Table 3.6. Mount the CryoProbe

step	action
m.1	Terminate any NMR experiment and remove sample and probe.
m.2	Set all RF power levels <i>p1</i> and <i>sp</i> to 120 dB (recommended).
m.3	Lower the magnet air suspension.
m.4	Inspect the Mounting Hardware: All four guiding rails must be in place and fixed tightly (two long vertical bars in the rear and two short ones in front).
m.5	Prepare the CryoProbe support plate: all five screws should be moved almost out of the plate.
m.6	Verify without a CryoProbe that the support plate will snap into the notches on the rails (Figure 3.4.). Remove it afterwards again.

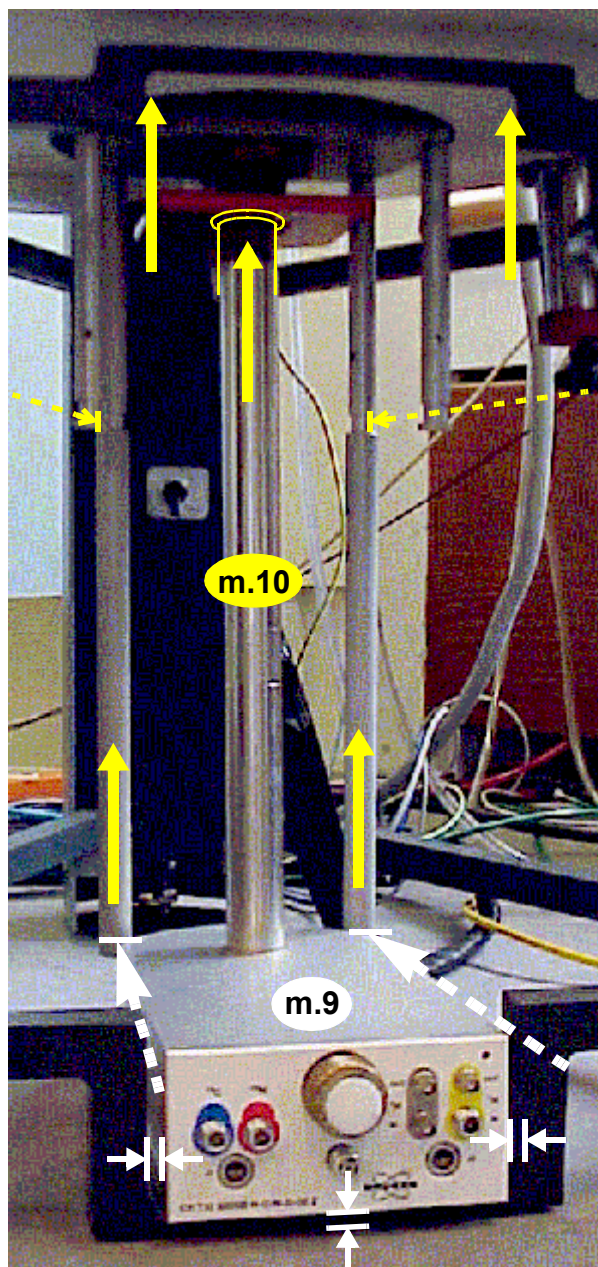
Figure 3.4. Preparing the Mounting Hardware



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

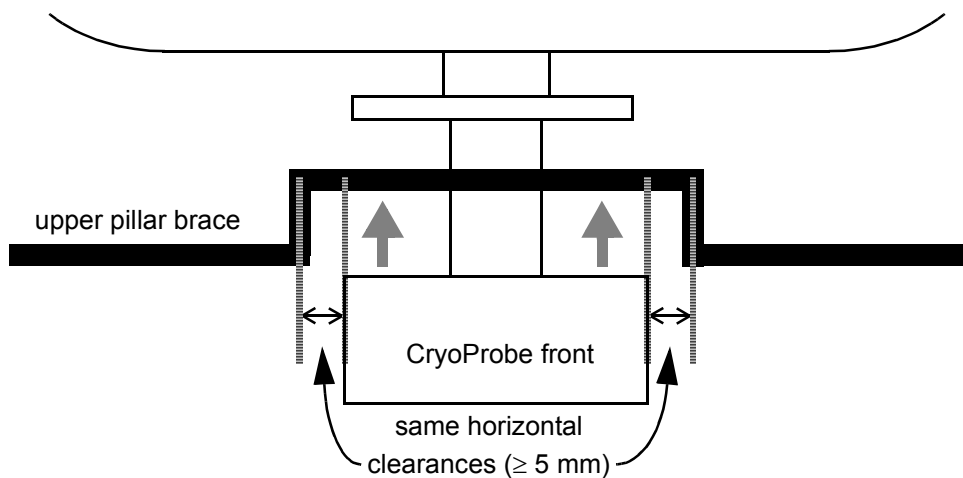
m.9	<p>WARNING: While mounting the CryoProbe, the magnet must not be suspended on its anti-vibration feet.</p> <p>WARNING: Do not attempt to insert or remove the heavy CryoProbe (~12 kg) at the magnet without the help of a second person. You may easily injure your back when lifting the CryoProbe on your own!</p> <p>Remove the protective cap from the top of the CryoProbe.</p> <p>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 3.5). Let its body touch the two long guiding rails that extend downward from the Mounting Hardware.</p>
m.10	<p>Align the CryoProbe top with the shim system bore.</p> <p>Push it gently up into the magnet until the support plate can be snapped into the notched guiding rails.</p> <p>Snap the support plate into place. Verify correct seat of all four support points.</p> <p>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.</p>

Figure 3.5. Sliding the CryoProbe into the magnet



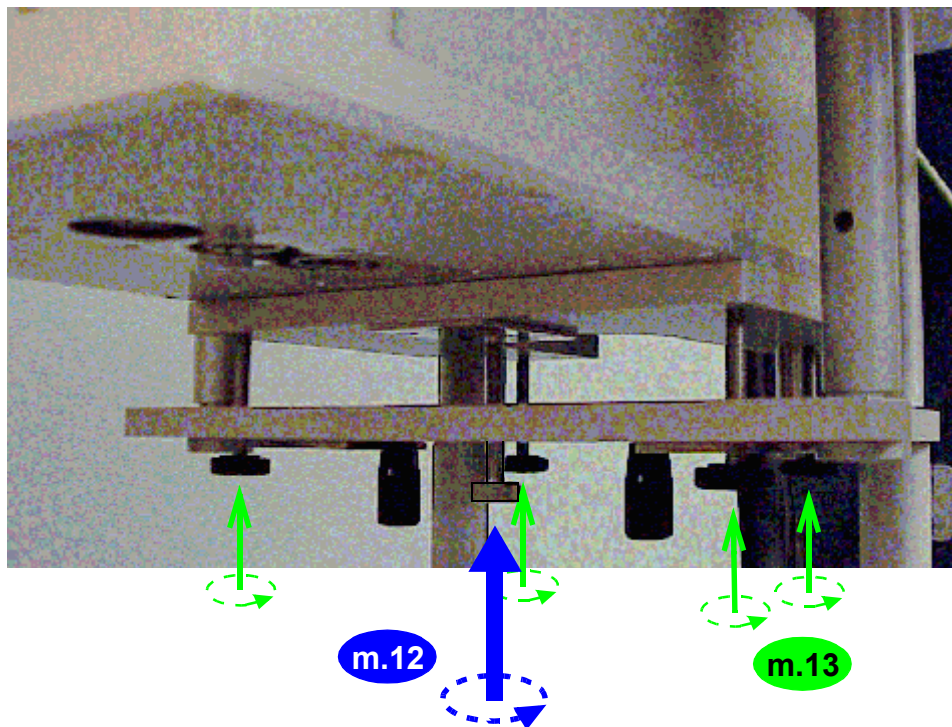
m.11	<p>Check the orientation of the CryoProbe in the magnet: Its front must be able to meet the He Transferline.</p> <p>IMPORTANT: If cranked pillar braces are present in the magnet stand, the CryoProbe must be centered in their gap at all times (see Figure 3.6).</p>
------	-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------

Figure 3.6. CryoProbe and optional cranked pillar braces



m.12	Push the CryoProbe up gently by turning the screw below its center until the CryoProbe body almost touches the shim system (gap \approx 0.5 mm). Feel for the end position, but upon reaching it, do not use great force to tighten the screw (Figure 0.4.).
m.13	Turn the other four screws on the support plate in an alternate fashion such that they support the CryoProbe gently. Take care not to tilt its body by forcing one screw more than the other screws.

Figure 3.7. Final positioning of the CryoProbe



Sample spinning test

3.11

Lift the magnet

CAUTION: If the CryoProbe is not correctly aligned, its body might touch the optional horizontal pillar braces when the magnet is lifted by about 10 mm onto its anti-vibration stand. The forces involved can easily break the CryoProbe! Before the magnet is lifted, there should be a symmetric **horizontal clearance of ≥ 5 mm** between the CryoProbe and any fixed obstruction above its body (see [Figure 3.6.](#)).

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!

Check the magnet alignment

Check the magnet's vertical position with a level and correct if necessary.

Spinning test

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

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

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

If spinning does not work, see "**Spinning problems**" on page 60.

Joining the CryoCoupler

3.12

During a CryoPlatform installation, the He Transferline usually needs to be slightly twisted before the CryoCoupler can be introduced smoothly into the CryoProbe.

CAUTION: If this adjustment is ignored, the He Transferline can exert a considerable torque on the fragile joint between CryoProbe body and tube.

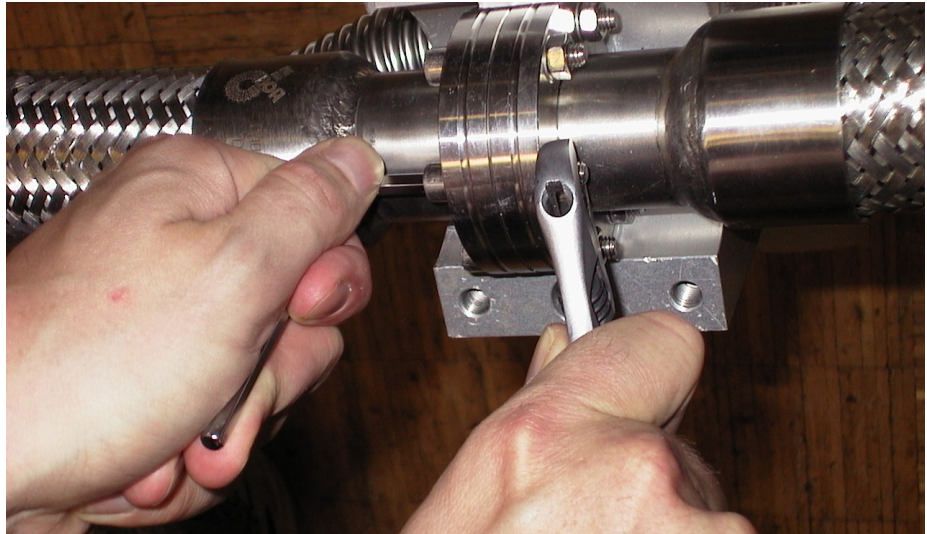
Since the twist angle depends on the relative positions of CryoCooling unit and magnet, the He Transferline must be fine-tuned on site after the CryoCooling Unit and the Transferline Support found their final positions. The same adjustment is needed whenever the relative positions of magnet and CryoCooling Unit have changed.

IMPORTANT: Do not try to align the CryoCoupler when cold. Do not open any screw other than those specified.

Table 3.7. Join the CryoCoupler

step	action
j.1	Detach the He Transferline from the Transferline Support but let it rest on the column.
j.2	IMPORTANT: Do the necessary adjustments only at the second vacuum flange when seen from the CryoProbe end - it features a special seal. Unlock all screws around the vacuum flange just a little and break the vacuum in the He Transferline. <u>Figure 3.8.</u>

Figure 3.8. Unlock the vacuum flange screws



j.3	Remove the protective caps from the CryoCouplers on He Transfer-line and CryoProbe.
j.4	Check the four o-rings on the He Transferline CryoCoupler (Figure 3.9): are they in place, clean and undamaged? If not replace with the o-rings found in the spare parts box delivered with each CryoProbe System. The o-rings are of type viton and size 7.1 by 1.6 mm.

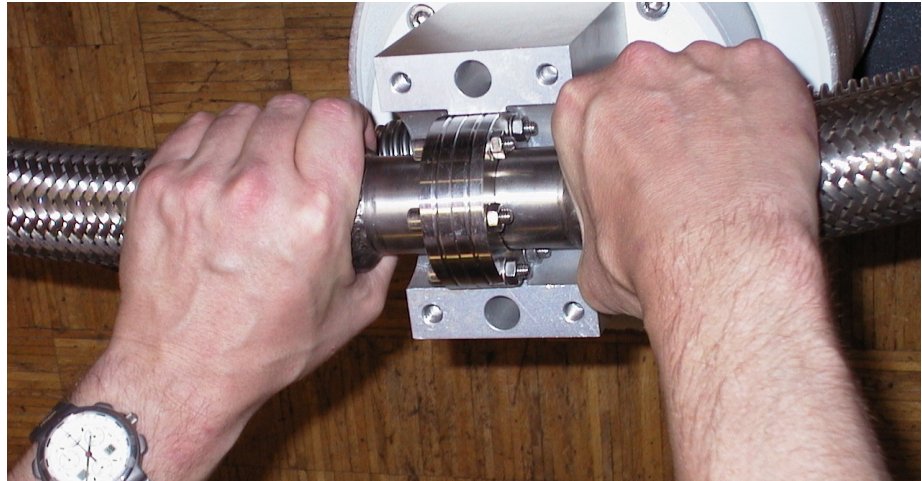
Figure 3.9. O-rings on the CryoCoupler



Initial setup

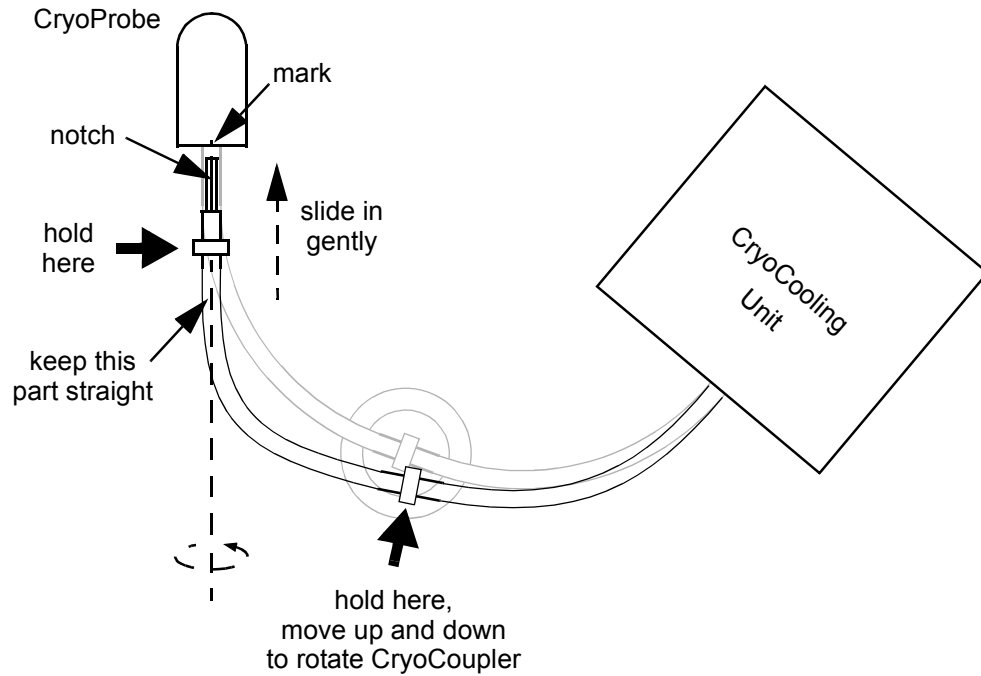
- | | |
|-----|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| j.5 | <p>Turn the CryoCoupler end such that it will fit into the CryoProbe: the groove must be exactly on top. Hold the Transferline strait to make the twisting smoother. <i>Figure 3.10.</i></p> <p>NOTE: No twisting should be necessary to insert the CryoCoupler into the CryoProbe. Do not re-tighten the vacuum connection on the He transferline yet.</p> |
|-----|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|

Figure 3.10. Turn the CryoCoupler



- | | |
|-----|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| j.6 | <p>Hold the CryoCoupler on the He Transferline with one hand. Take the vacuum joint which is 1 m away from the CryoCoupler into the other hand (see <i>Figure 3.11.</i>).</p> |
|-----|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|

Figure 3.11. Inserting the CryoCoupler



j.7	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.8	<p>IMPORTANT: It may be necessary to fine-tune the position of the Cryo-Cooling Unit before the connection can be established. Do not pull the He Transferline into position, its internal structure is fragile!</p> <p>Slide the CryoCoupler into the CryoProbe. It should fit smoothly without any significant movement of the suspended magnet.</p> <p>CAUTION: If the CryoCoupler gets stuck, do not force it into position. Do not bend the He Transferline to an extreme shape to make it fit - permanent damage on CryoProbe or He Transferline could result!</p> <p>The He Transferline Support may be in the way during the insertion process. If this is the case, remove the CryoCoupler, move the Transferline Support a little aside and repeat the insertion process.</p> <p>When a smooth insertion fails, take the CryoCoupler out and try again.</p>

j.9	<p>If the insertion is working smoothly tighten all screws on the vacuum flange very carefully in a balanced fashion to align the seal inside properly.</p> <p>NOTE: If a subsequent cool-down fails due to vacuum problems, the vacuum flange may not have been closed properly.</p>
j.10	<p>Use the special open-end titanium wrench to fix the CryoCoupler gently to the CryoProbe. Take care not to damage any connectors on the CryoProbe front. Hold the He Transferline end with your other hand to take up the torque applied.</p>
j.11	<p>Position the Transferline Support such that the He Transferline falls exactly (to within 1 mm) into its bed.</p>
j.12	<p>Make sure that the CryoProbe is still centered inside the (optional) cranked pillar braces. If not, relocate the Transferline Support accordingly.</p> <p>NOTE: Ensure that the He Transferline bending and the Transferline Support position are the same as they will be for the operation of the CryoProbe. If feasible, mark the location of the Transferline Support on the floor.</p>
j.13	<p>Lock the He Transferline on the Transferline Support with the four black screws.</p>

When installing only an additional CryoProbe on a working CryoPlatform, continue with **"Connecting" on page 46.**

Load firmware

3.13

A laptop is delivered for monitoring purposes that has the necessary software and directory structure pre-installed. If the versions of *UniTool* and *CryoController Firmware* are not the most current ones, they need to be updated.

Directory structure

On the CryoProbe System laptop, the following directories are required:

D:\app\Bruker\unitool	<i>UniTool</i> program
D:\app\Bruker\unitool\files\crco	<i>Firmware</i> file crcoxx.abs
D:\app\Bruker\unitool\files\temp	
D:\app\Bruker\CryoTool	<i>CryoTool</i> program
D:\app\Bruker\CryoTool\LogFiles	logfiles

Users

An `Administrator` with the password `password` and a `Power User` `cryouser` without password protection are enabled.

Get the software releases

Connect to the BRUKER *ftp.bruker.ch* server and login either as *ftp* or *anonymous* with your full e-mail address as password.

Transfer the *UniTool* release files *unitool.** and *setup.exe* from */pub/NMR/download/servtools/pc/unitool* to the local directory *D:\app\Bruker\unitool*.

Similarly, transfer the *Firmware* distribution *Crcoxx.abs* (*xx* being a code for the version) from */pub/NMR/download/servtools/firmware/crco* to the local directory *D:\app\Bruker\unitool\files\crco*. Make sure that only one file called *Crcoxx.abs* resides there.

The *CryoTool* software cannot be downloaded from a public server, it must be requested from BRUKER.

UniTool installation

Before installing a new UniTool version, prevent conflicts with previous versions by either renaming the old files or using the specific *uninst.exe* program provided with the original distribution which can be accessed e.g. via **Start - Programs - UniTool - Uninstall UNITool**.

Read the corresponding *readme.txt* file first. Execute the corresponding *setup.exe* file. When asked for the *UniTool* installation directory, specify *D:\app\Bruker\unitool*. The *LabWindows/CVI Run-Time Engine Directory* should be the default *D:\WINNT\System32\CVIRTE*. Click **Finish**.

Firmware download

Read the corresponding *readme.txt* first. Start the *UniTool*, specify **COM1** and select **[2] Check/Download CRCO** from the main menu. All lights on the Cryo-Cooling Unit will fade except for *ERROR* which will blink slowly (8 sec on - 8 sec off). The download will transfer the *Crcoxx.abs* file from *D:\app\Bruker\unitool\files\crco* to the CryoController. It lasts for a couple of minutes and ends with an automatic reboot of the CryoController.

Shortcuts

There are both desktop icons and start menu entries for **CryoTool**, **UniTool**, and **CryoTool LogFiles**.

The working directory of the *CryoTool* must be set properly such that all logfiles are written automatically to the correct location: right-click **Start - Explore** - select *D:\WINNT\Profiles\All Users\Desktop\Bruker* - right-click **CryoTool - Properties - Shortcut - Start in:** *D:\app\Bruker\CryoTool\LogFiles*. Repeat this procedure for *...\Start Menu\...* instead of *...\Desktop\...*

Boot the CryoController

3.14

CAUTION: Lay all tubes and cables such that they don't cross the floor on a walkway. If a crossing cannot be avoided, burry or cover them.

NOTE: There is no point in testing components of the CryoProbe System separately. Fault analysis is easiest when the CryoProbe System is tested as a whole

because most procedures are fully automated and supervised by the CryoController.

Table 3.8. *Booting the CryoController*

step	action
u.1	Remove all panels from the CryoCooling Unit (see <u>"How to open the CryoCooling Unit" on page 67</u>).
u.2	Attach pneumatic gas of at least the minimum specified pressure to the <i>Comp. air</i> or the input at the rear of the CryoCooling Unit. Check the gauge inside the CryoCooling Unit and adjust the compressed air pressure if necessary. CAUTION: The compressed air supply must never be turned off.
u.3	Put the laptop either onto the CryoCooling Unit, next to the workstation, or on the spectrometer cabinet but keep it outside the 1.0 mT range of the magnet. NOTE: The laptop can be supplied with 100-240 V, 1.5 A, 50/60 Hz. Most convenient is one of the 'Schuko' sockets on the inside of the CryoCooling Unit although they are switched with the mains. The laptop has an internal battery pack and is thus fairly independent from the CryoCooling Unit.
u.4	Plug the external mouse into the laptop. WARNING: The laptop features also a touch pad and a pointing stick. These devices must not be used in the CryoTool's <i>Service</i> mode. They can easily lead to erroneous clicks with fatal results when operating e.g. individual valves.
u.5	Connect the CryoController's RS232 output (located inside the CryoCooling Unit) to <i>COM1</i> port of the laptop (labelled <i>IOIO</i> on its back) with the 9-pin computer cable Z14126. IMPORTANT: The CryoController's RS232 interface must not be used for other purposes!
u.6	Boot the laptop and login as cryouser (no password required).
u.7	Link the BVT cable from the VT Interface Box to the BVT socket on the CryoController inside the CryoCooling Unit.
u.8	Extent the other line from the VT Interface Box with cable Z14278 which will go to the CryoProbe .

u.9	<p>Plug-in the CryoCooling Unit mains.</p> <p>IMPORTANT: Do not supply the CryoCooling Unit from the spectrometer backpanel because after a power failure an AVANCE cabinet and its auxiliary mains outputs remain <i>OFF</i>.</p> <p>NOTE: A supply socket must have been installed already during the site preparations. If no standard IEC 320 C4 socket (= German 'Schuko' two-pole plug with dual earthing-contacts, rated 10/16 A 250 V) is available, it can be replaced by a connector type that complies with the local regulations for 230 V and 500 W/1500 W average/peak power. Wire assignments are: brown = line ('field'), blue = neutral ('field'), yellow/green = ground.</p>
u.10	<p>Turn the rotary main switch at the CryoCooling Unit front ON. The Cryo-Controller will boot automatically.</p> <p>NOTE: If its buttons don't light up after a second, check the automatic circuit breakers on its backpanel and the 50 mA fuse. Also, there must be a dummy plug on the <i>REMOTE SWITCH</i> socket which short-circuits two pins.</p>
u.11	<p>Start the <i>CryoTool</i> on the laptop by either clicking its desktop icon or Start - Bruker - CryoTool - CryoTool. Select <i>COM1</i>, double-click the '<i>F</i>' in the word <i>Firmware</i> with the right (!) mouse button, and click <i>Service</i> to bring up the full interface and <i>Log</i> to see the system messages.</p> <p>NOTE: If the CryoTool is started before the CryoCooling Unit is switched <i>ON</i>, it will go to off-line mode because it sees no response at its <i>COM1</i> port.</p>

Hook up the He steel-cylinder

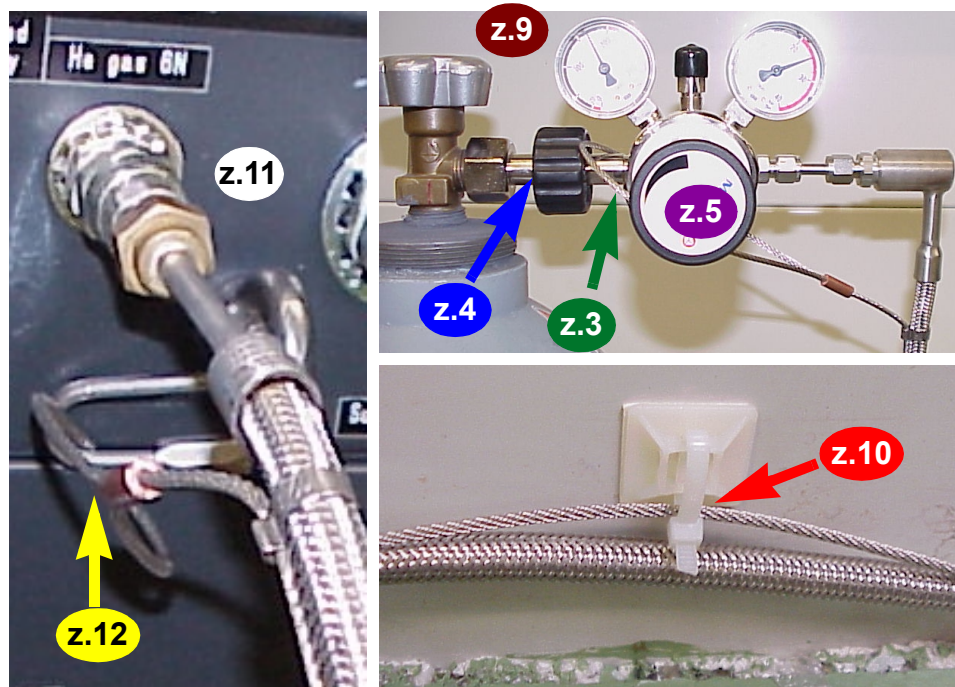
3.15

WARNING: Wear protective goggles. Keep away from directions into which components the pressurized system could be expelled in case of an accidental rupture.

Table 3.9. Mount the He steel-cylinder

step	action
z.1	<p>Check if the He steel-cylinder is of Grade 6.0 (i.e. 99.9999% purity, 'electronic' grade). Move it into position and fix it tightly to a wall.</p> <p>WARNING: The He steel-cylinder and its entire transport path must always be outside the 0.5 mT range of the magnet.</p>
z.2	Remove the protective cap from the He steel-cylinder.
z.3	Pull the steel cable loop over the pipe from the He Regulator to the He steel-cylinder (Figure 3.12).

Figure 3.12. Attaching the He Hose



z.4	Make sure that the pipe interfaces are clean and screw the He Regulator tightly to the He steel-cylinder.
z.5	The He Regulator valve must be completely closed .
z.6	Open the He Regulator by about one turn and connect the dump tool to the other end of the He Hose.
z.7	Open the He steel-cylinder main valve . The primary pressure gauge will rise up to 200 bar and the sound of escaping He from the dump tool will become audible.
z.8	Close the He Regulator and disconnect the dump tool. IMPORTANT: The He Regulator and Hose have to be flushed to keep impurities out of the He steel-cylinder.
z.9	<u>1st leak test</u> : mark the actual pressure on the primary gauge and close the He steel-cylinder main valve.

z.10	<p>Lay the He Hose to the CryoCooling Unit such that it does not cross the floor on a walkway. If a crossing cannot be avoided, bury or cover the He Hose.</p> <p>Attach the He Hose steel wire at intervals of about 1 m firmly to the wall or to the floor.</p> <p>WARNING: Avoid the use of magnetic materials or tools inside the 0.5 mT stray field of the magnet.</p>
z.11	<p>Make sure that connector on the He Hose is clean before connecting it to the CryoCooling Unit back panel. Use two wrenches at the hose's two terminal hexagon nuts for attaching it. When a tight connection has been established, unlock the connectors by ~1/8 turn to facilitate later changes.</p>
z.12	<p>Lock the steel cable loop in the hook that is provided at the CryoCooling Unit backpanel.</p>
z.13	<p><u>End of 1st leak test:</u> Check the pressure on the He Regulator's primary gauge. If the pressure has dropped in the meantime, the connection between He Regulator and He steel-cylinder needs tightening.</p>
z.14	<p>Open the He steel-cylinder main valve by ~one turn.</p>
z.15	<p>Open the He Regulator slowly such that a secondary pressure of 22-25 bar builds up. Do not overshoot this pressure. The pressure cannot be reduced by closing the He Regulator because the He cannot escape.</p> <p>IMPORTANT: Never exceed 30 bar - the gauge would be damaged!</p> <p>If 25 bar were exceeded significantly, close the He Regulator. Disconnect the He Hose from the CryoCooling Unit and terminate it with the Dump Tool to release the trapped He. Re-connect the He Hose to the CryoCooling Unit and repeat step z.15.</p>
z.16	<p><u>2nd leak test:</u> Close the He steel-cylinder main valve. Mark the secondary pressure on the gauge and check again later. Within the first ½ h, the pressure might drop a little but it must not decrease thereafter.</p> <p>NOTE: The 2nd leak test will be terminated in <u>"Interconnect the He Compressor" on page 44</u>, step <u>i.12</u> below.</p>

Interconnect the He Compressor

3.16

The following section was partially adapted from the He Compressor operation manual which is delivered with the CryoPlatform.

CAUTION: Lay all tubes and cables such that they don't cross the floor on a walkway. If a crossing cannot be avoided, bury or cover them.

Necessary tools:

Initial setup

- 1 open-end wrench 1-³/₁₆"
- 1 open-end wrench 1-³/₈"

Table 3.10. Interconnect the He Compressor

step	action
i.1	<p>IMPORTANT: The o-rings and rubber gaskets on all Flexline connectors must be very clean and properly positioned.</p> <p>Use a flashlight for inspecting the Flexline connectors and their counterparts on the He Compressor and the CryoCooling Unit. If necessary, clean them with a tissue and tweezers but do not use aggressive solvents.</p>
i.2	<p>CAUTION: The attachment order of the Flexlines is important: Always start with the green RETURN line (i.e. at the low pressure side of the gas cycle), then connect the yellow SUPPLY line. Connect them first at the He Compressor.</p> <p>Loosely attach the Flexline with the green RETURN marker to the GAS RETURN port on He Compressor. Each Flexline is symmetric, i.e. there is no preferred orientation.</p> <p>CAUTION: Do not interchange SUPPLY and RETURN. The Coldhead would become inoperable when switched on and can only be repaired through complicated service procedures!</p>
i.3	<p>When locking the green RETURN Flexline, make the first turns by hand. Then, use open-end wrenches 1-³/₁₆" and 1-³/₈" on the Flexline's two terminal hexagon nuts. After reaching the stop, rotate back by a ¼ turn to facilitate eventual unlocking.</p> <p>Treat the yellow SUPPLY Flexline similarly.</p> <p>NOTE: Do not put any force on the Flexlines third hexagon nut which ties the connector to the actual tube. The minimum bending radius of a Flexline is 0.3 m, i.e. a 180° turn has a diameter of at least 0.6 m! Do not twist the Flexlines when making the final connections. Do not untie the hexagon nuts on the He Compressor but check if they are moving while the Flexlines are fixed. If they are loose, tighten them to the He Compressor housing before proceeding with the Flexlines.</p>
i.4	<p>Fix the green RETURN Flexline tightly to the CryoCooling Unit's RETURN port. Mount the yellow SUPPLY last.</p>
i.5	<p>After connecting all Flexlines, check the SUPPLY PRESSURE gauge on the He Compressor front. It should read a bit more than 0 bar without a drop during several minutes.</p> <p>IMPORTANT: If the pressure gauge indicates plain 0 bar, contact BRUKER: this is a major problem and a lengthy gas purifying cycle is required.</p>

i.6	<p>If the He Compressor requires water-cooling, connect it to the in-house cooling water cycle or to the optional water chiller.</p> <p>NOTE: The installation of the water cooling system is not subject of this manual.</p> <p>Turn on the water and check for leaks.</p>
i.7	<p>If the He Compressor was not already hooked-up to its 3-phase mains, check that the MAIN POWER SW on the He Compressor back and DRIVE on the front are OFF before establishing this connection.</p>
i.8	<p>Plug the 4-pin Coldhead power cable into COLD HEAD POWER on the CryoCooling Unit and COLDHEAD JC on the He Compressor.</p> <p>CAUTION: Never connect the Coldhead cable between He Compressor and Coldhead while the He Compressor is energized.</p>
i.9	<p>Connect the cable Z13883/Z14323 from the CryoCooling Unit to EXTERNAL JR on the He Compressor. Set the REMOTE DRIVE at the He Compressor back to EXT position.</p> <p>CAUTION: The COLDHEAD DRIVE switch is for maintenance purpose only and must be OFF at all times. Do not touch it during normal operation. An accidental switch to ON may cause severe damage to the Coldhead within a few hours.</p>
i.10	<p>Note down the values from the hour meters on the He Compressor front and the CryoCooling Unit back.</p>
i.11	<p>CAUTION: Ensure that the MAIN POWER and DRIVE switches on the He Compressor back and front, respectively, are OFF before connecting the He Compressor unit to mains.</p> <p>Plug-in the He Compressor but do not switch it ON yet.</p>
i.12	<p><u>End of 2nd leak test:</u> Check on the He Regulator low pressure gauge if the secondary pressure has remained stable, i.e. if there is no leakage between the He Regulator and the He entry valve inside the CryoCooling Unit.</p>
i.13	<p>Open the He steel-cylinder main valve by ~one turn.</p>

Warning labels

3.17

Stick a warning label (**Figure 3.13.**) on all mobile units of a CryoProbe System that contain magnetic materials and could be attracted by the NMR magnet.

Initial setup

Put a label on or next to the holder of the He steel-cylinder. If the He Compressor or the optional water chiller are in the same room as the NMR magnet, label them also. They are on wheels and therefore potentially mobile.



Figure 3.13. 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!

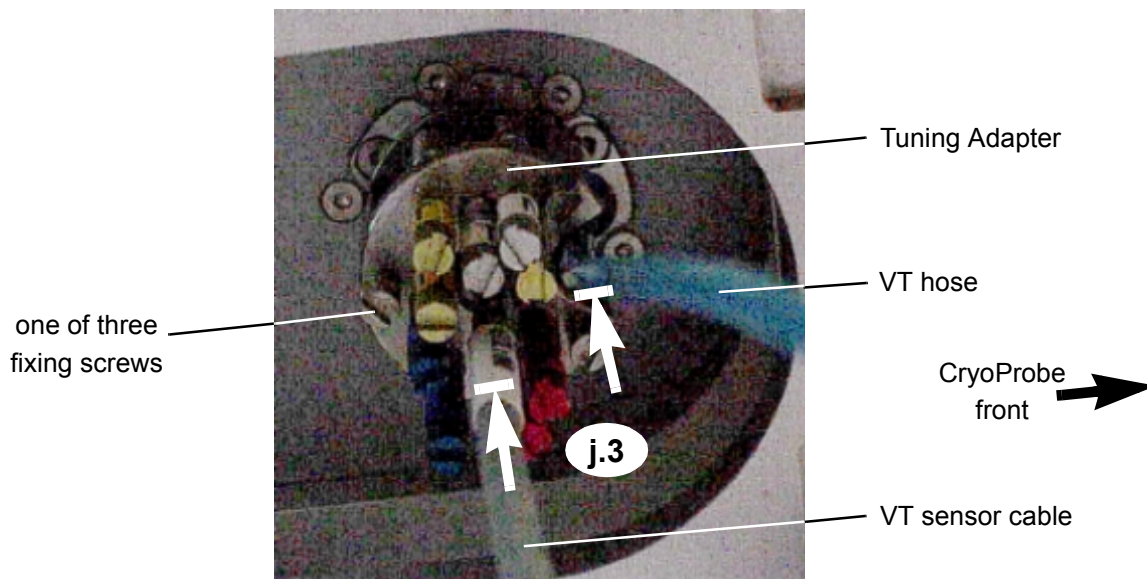
Connecting

3.18

Table 3.11. Connect the CryoProbe

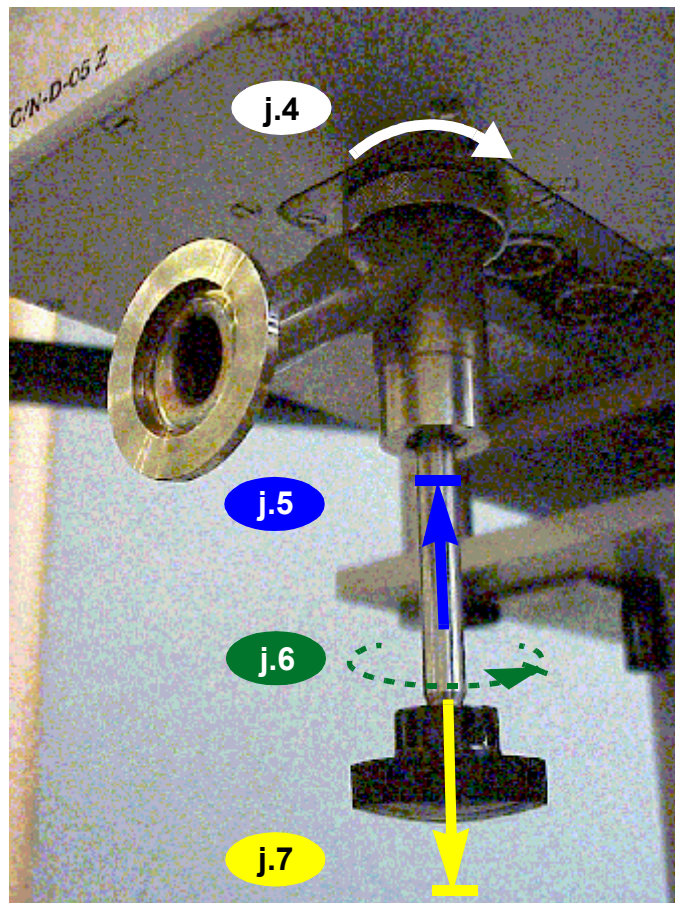
step	action
j.1	Plug the two sensor cables from the CryoCooling Unit into the CryoProbe bottom and into J2 on the front, respectively. Connect the Cryo-Preamplifier supply cable from the HPPR CRP to J1.
j.2	Connect the VT sensor cable and the VT gas hose to the probe bottom. Check if all connections for the VT Interface Box are properly made. At the VT unit, e.g. B-VT3000, the Pt100 sensor must be selected. The reading in <i>edte</i> should indicate room temperature .
j.3	Attach the Tuning Adapter (Figure 3.14.) to the CryoProbe bottom. Use the blue Tuning Tool to lock its mounting screws. NOTE: There are specific Tuning Adapters for each type of CryoProbe.

Figure 3.14. Connecting VT sensor cable and VT hose (bottom view)



j.4	Mount the Vacuum Adapter to the CryoProbe bottom such that it points to the front. Connect the vacuum tube .
j.5	Push in the actuator screw of the Vacuum Adapter (black handle) in as far as it will go (Figure 3.15).
j.6	Turn the actuator screw clockwise . Its thread must grip into the thread of the CryoProbe's vacuum plug. Continue gently until the stop is reached but do not force it into the stop.

Figure 3.15. Open the CryoProbe vacuum plug



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

j.11	Switch the VT heater power <i>ON</i> .
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Power-on**3.19**

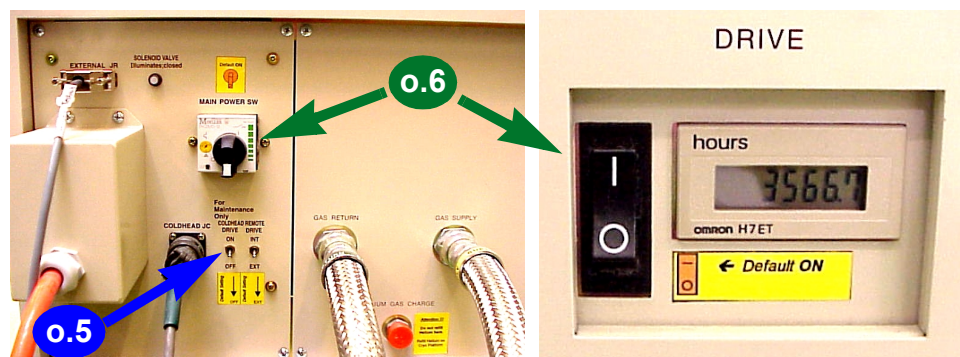
If the CryoProbe System is *OFF* or has not been used immediately before, go through the following steps to make sure that all components are *ON* and ready.

CAUTION: Before powering-up a CryoProbe System for the first time, e.g. during an installation, double-check if the voltages and fuses in the laboratory meet the requirements given on the backpanels of CryoCooling Unit, He Compressor, and (optional) water chiller.

Table 3.12. *Power-on*

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

Figure 3.16. Switching on the He Compressor



VT setup

3.20

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

See "[Sample temperature control](#)" on page 55 in the *User Manual* for details on the sample temperature setup.

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

Both N₂ gas and dry air can be used as VT gases. For a CryoProbe, there is no difference between them with respect to shimming or RF performance.

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

Table 3.13. VT setup

step	action
v.1	<p>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 <i>On</i> yet.</p> <p>CAUTION: When cold operation has been started, the VT gas supply must not be interrupted before the CryoProbe has been warmed-up again. Thus, a BCU05 gas cooler can only be inserted while the CryoProbe is warm.</p>
v.2	<p>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 <i>on</i> yet.</p> <p>NOTE: A high VT gas flow rate is recommended but it must not lift the sample.</p>
v.3	Verify that there is no sample in the magnet yet .
v.4	<p>Configure the VT unit for the Pt100 temperature sensor with <i>edte</i>. Make sure the Pt100 sensor cable is connected properly at CryoProbe and VT unit.</p> <p>NOTE: An optional B-TO2000 unit for conventional probes uses the same type of Pt100 cable. The cable is suitable for the CryoProbe and there are no special considerations when using a B-VT3000. A B-VT2000, however, cannot be toggled properly from B-TO2000 to CryoProbe operation with <i>edte</i> (although no error occurs); it must be set explicitly at its EURO THERM module. With a B-TO2000, the EURO THERM 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 EURO THERM is configured for Pt100 with code 0225. See the <i>B-VT2000 Operating Manual</i> (P/N W1101034) for instructions.</p>
v.5	Set the sample temperature to somewhere above room temperature, e.g. 300 K .
v.6	Switch the VT heater <i>on</i> .
v.7	Wait until the 'sample' temperature and the heater power in the <i>edte</i> window settle.

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

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

The He Compressor is usually shipped almost pressure-less. Thus, if its *SUPPLY PRESSURE* gauge reads significantly less than 16 bar (= 1.6 MPa or 16.3 kgf/cm²), it must be filled from the He steel-cylinder via the CryoCooling Unit, see "**Refill He**" on page 67.

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

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

Table 3.14. Cool-down

step	action
c.1	<p>Check if the</p> <ol style="list-style-type: none"> 1) cooling water supply is active (if applicable); 2) He steel-cylinder is connected and open. Its primary pressure must be larger than 40 bar and its secondary pressure stable at 22-25 bar; 3) CryoProbe is properly installed and connected; 4) CryoCooling Unit is <i>ON</i> and if WARM and unplug indicate that the unit is ready; 5) <i>SUPPLY PRESSURE</i> gauge on the He Compressor front reads about 17.5 bar (= 1.75 MPa or 17.9 kgf/cm²). If the pressure is higher, the excess He will be released automatically during the cool-down preparations. If the pressure is much lower than 16 bar (= 1.6 MPa or 16.3 kgf/cm²), see <i>Refill He</i> in the <i>Installation Manual</i>; 6) He Compressor MAIN POWER SW at its backside and <i>DRIVE</i> at its front are ON.
c.2	<p>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.</p>
c.3	<p>Verify that there is no sample in the magnet yet.</p>
c.4	<p>Start the cool-down by pushing the <i>COOL DOWN</i> button on the CryoCooling Unit or in the <i>CryoTool</i>. <i>COOL DOWN</i> will start to flash, indicating that the system is preparing the cool-down process.</p>

c.5	<i>COOL DOWN</i> will keep flashing as long as the ambient temperature preparations (evacuation, flushing cycles and charging with He) are in progress. Several pneumatic pops will be audible. At this point, it is recommended to check the secondary pressure at the He Regulator: it should always come back to 22-25 bar . The primary pressure must remain ≥ 40 bar at all times.
c.6	~15 min later, CryoCooler and He Compressor will be started automatically to perform the cool-down. <i>COOL DOWN</i> changes to steady white during the actual cool-down. The characteristic periodic hiss of the CryoCooler will be audible then.

When the cool-down has started, **"Load additional Xwin-nmr software" on page 54** and **"Put HPPR CRP assembly into operation" on page 54**. Do not leave the CryoProbe System unattended during the first cool-down.

While the cool-down is in progress for about 2 h, there is plenty of time to do the **"HPPR CRP configuration" on page 45** in the *User Manual*.

c.7	<p>If COLD on the CryoCooling Unit flashes, it will take about half an hour more until the final temperature is reached and stabilized.</p> <p>NOTE: A cool-down must not take more than 4 h; if it does, the Cryo-Controller will interrupt automatically and warm up the system (see <u>"Cool-down doesn't reach cold state" on page 66</u>).</p>
c.8	<p>Verify that the 'sample' temperature reading from inside the cavity is at its preset value and stable. Now a sample can be inserted. At this point, the <u>"Low temperature limit in sample cavity" on page 46</u> in the <i>User Manual</i> should be determined.</p> <p>NOTE: For recommended sample depth and filling height, see the 'LIMITATIONS - WARNINGS' sheet for the CryoProbe and <u>"Samples and spinners" on page 55</u> in the <i>User Manual</i>.</p> <p>IMPORTANT: Always keep an eye on the sample temperature with <i>edte</i> when working with the CryoProbe. If the temperature drops, eject the sample and keep monitoring the 'sample' temperature inside the CryoProbe cavity.</p>
c.9	Set all RF power levels <i>p1</i> and <i>sp</i> to 120 dB (recommended).
c.10	Enter <i>xau crpwobb</i> in XWIN-NMR and tune the CryoProbe on all channels including ^2H , see <u>"Tuning and matching" on page 48</u> in the <i>User Manual</i> . When done, enter <i>xau crpon</i> .
c.11	Shimming can be started while <i>COLD</i> is still flashing, see <u>"Shimming and Lock-in" on page 49</u> in the <i>User Manual</i> .
c.12	When COLD is continuously on , the final conditions are stable.

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

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

Load additional XWIN-NMR software

3.23

Install the AU-programs *crpon*, *crpoff* etc. for preamplifier selection on the spectrometer workstation. This can be done conveniently while the first cool-down is in progress.

Get the software release

Connect to the BRUKER *ftp.bruker.de* server and login either as *ftp* or *anonymous* with your full e-mail address as password.

Transfer the *crponoff.** files from */pub/nmr/XWINNMR/utilities/* to the directory on the spectrometer workstation where XWIN-NMR is installed (usually */u* under UNIX and *C:\Bruker\XWIN-NMR* under Windows NT).

UniTool installation

Read the *crponoff.readme* file first. Unzip the *crponoff.tar.gz* file and unpack the resulting *.tar* file. In XWIN-NMR, execute *xau crpon* to compile the new AU-program *crpon*. Do the same for *crpoff*, *crpwobb*, *crp1hxon*, and *crp2hon*.

Put HPPR CRP assembly into operation

3.24

NOTE: The HPPR CRP can be installed conveniently while the CryoProbe undergoes its first cool-down.

The HPPR CRP configuration specifically matches the frequency channels of the actual CryoProbe. Usually, a conventional X-BB 19F 2HS is included to permit operation with arbitrary conventional probes.

Example: CryoProbe TXI H-C/N-D (see also *User Manual Figure 4.14*)

HPPR: 1H CRP
X-BB 19F 2HS
2H CRP
13C CRP
15N CRP

An HPPR CRP can supply 5 preamplifier modules at most. If more modules are required to suit the user's needs, their wiring must be checked during the setup of each NMR experiment - and manually changed when necessary.

Table 3.15. Installing the HPPR CRP

step	action
e.1	Shut down the HPPR's power supply by switching OFF the AQR unit inside the spectrometer cabinet.

e.2	If some conventional HPPR modules are to be kept, merge them into the HPPR CRP but do not connect any cables to them yet (see <i>CryoProbe RF Electronics Technical Manual</i> for details).
e.3	Check the ribbon cables and the coax cables at the HPPR CRP side: are they configured to match the frequency channels of the actual CryoProbe? Do not connect cables of other HPPR modules to the cover module (e.g. do not connect the conventional X-BB module when using a CryoProbe TXI).
e.4	Check the jumper setting in the existing HPPR cover module and establish the same configuration in the new HPPR CRP. Default jumper settings are listed on a sticker inside the cover. NOTE: If jumper <i>JU2</i> is encountered in the non-default position on the existing HPPR, it is likely that the corresponding jumper <i>JU20</i> in the RX22 receiver is also in its non-default position for some reason. Both jumpers affect the polarity of the RGP (also called 'EP') trigger signal.
e.5	Check also the jumpers at the side of the HPPR CRP modules for X-nuclei: e.g. at the 13C CRP module for a CryoProbe TXI, a jumper should bridge the upper two pins (labelled <i>17</i> and <i>18</i>); whereas at the 15N CRP module the lower two pins should be connected (<i>18</i> and <i>19</i>).
e.6	Switch On the AQR and run an <i>XWIN-NMR cF</i> dialogue. The first X-module in the HPPR CRP will be recognized as <i>X-BB31P_2HS</i> and an optional second module as <i>X-BB19F_2HS</i> . If more than 2 modules for X-nuclei are active, the <i>cF</i> dialogue may run into an error.
e.7	Run <i>xau crpwobb</i> in <i>XWIN-NMR</i> to configure the HPPR CRP for a wobbling test. Check the wobbling of each HPPR CRP module (including 2H) with an appropriate conventional probe.
e.8	When all tests of the required HPPR CRP modules were passed, swap the connections and test the other HPPR modules.

How to proceed

3.25

When the CryoProbe is cold and ready, continue in the *CryoProbe System User Manual* with **"HPPR CRP configuration" on page 44.**

This *Installation Manual* will be used again later for the installation of options and for customizing the system.

Customizing the CryoProbe System

4

Experimental parameters

4.1

RF power levels

4.1.1

Maximum RF powers are specified in Watt for each CryoProbe on its 'LIMITATIONS - WARNINGS' sheet. Since the relation between absolute power and $p1$ values depends on the actual spectrometer hardware, the RF limits must be translated into power levels $p1$. If CORTAB is used on the spectrometer, determine them with the actual CORTAB and - for safety - without CORTAB. Write these $p1$ values down on the 'LIMITATIONS - WARNINGS' sheet and notify the NMR superuser.

Safety attenuators

4.1.2

If desired by the customer, insert RF attenuators before the inputs of the RF amplifiers such that their maximum output power at $p1=-6$ does not exceed the limits of the CryoProbe.

Standard parameter sets

4.1.3

Enter the CryoProbe into the *edhead* table and store its pulse parameters for use in routine operation or automation.

Lock power

4.1.4

In contrast to the 1H and X channels, the lock channel of a CryoProbe may need as much (or even more) RF power as in a conventional probe. Initiate an automatic *lock*, perform an AUTO LOCK POWER calibration with the BSMS keyboard, and store the result in the CryoProbe's *edlock* table.

Sample temperature control

4.1.5

Self-tune the VT unit (see *User Manual*, "[Tuning the regulation loop](#)" on page [57](#)).

When using a BCU05 gas cooler, care must be taken that the VT gas is not colder than the lower sample temperature limit (see LIMITATIONS - WARNINGS sheet)

Customizing the CryoProbe System

when entering the CryoProbe. Special care must be taken to avoid leaks such that the VT gas remains dry and its flow rate constant.

Sample tubes and spinners

4.1.6

A CryoProbe has smaller tolerances for sample spinning than conventional probes. Therefore, the spinning properties of a selection of typical customer sample tubes and spinners should be tested. Avoid cheap sample tubes or spinners with poor axial symmetry.

Receiver sensitivity

4.1.7

For small receiver gain values, i.e. $rg < \sim 256$, the signal-to-noise tends to decrease in proportion to rg . If the CryoProbe will be used mostly with samples that yield strong NMR signals like H₂O solutions, a reduction of the overall receiver gain may be beneficial because this decreases also the noise added by the receiver. The modification is a jumper setting in the RX22, ECL07 or later, according to the description given on the board itself. As a consequence, higher rg values than before will be required for all measurements.

CAUTION: Switch off the AQR and ensure ESD protection before manipulating the highly sensitive RX22 board.

Acceptance tests

4.2

Repeat the finaltests contained in the CryoProbe's customer documentation folder.

User training

4.3

See the checklist "**User training**" on page 76.

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

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

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

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

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

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

Fuses and reset buttons

5.1

CryoCooling Unit

Automatic fuses are located on the back panel along with a small melting fuse (European size, 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*.

CryoController

The CryoController is located inside the CryoCooler cabinet (see "**How to open the CryoCooling Unit**" on page 67). Its **primary fuses** (2 × 2.5 A slow) are located at its mains socket.

There are **secondary fuses** (2 × 10 A slow) on the main board close to the fan module. To check them, the CryoController case must be opened. Ensure ESD protection when working on an open CryoController!

He Compressor

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

HPPR CRP

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

General failure

5.2

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 Cryo-Cooling Unit front (this is the same as an **"" on page 59**).
3. Check the fuses.
4. Investigate the power supply; check all regulated voltage outputs.

No mains

5.2.1

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 59**.

Spinning problems

5.2.2

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

During operation

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

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

Eject the sample and check if it is ok.

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

After insertion of a new sample

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

Check the CryoProbe cavity for dirt and clean it if necessary (see **"Cleaning" on page 68** in the *User Manual*).

When the CryoProbe had been removed and was mounted again

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

If the gap is larger, do the five screws in the Mounting Hardware fail to support the CryoProbe? If they are ok, lower the CryoProbe by a few mm and re-insert it

again. If this doesn't help, inspect the CryoProbe top and the shim system for dirt or damage.

If there is no gap, check if the CryoProbe's circumferential top edge touches the blue spinner stator: take the CryoProbe out and put a soft non-magnetic ring onto its top, e.g. an o-ring or a ring made of cardboard. The ring should have a well-defined height and an outer diameter of less than 38 mm. Insert the CryoProbe. Measure the new gap and subtract the height of the ring. The missing length between CryoProbe and spinner stator results. If it is in the order of 1 mm or more, remove the shim system (see **"Replacing the shim system (optional)" on page 24**) and inspect it for dirt, misalignment, or damage.

IMPORTANT: Do not remove the blue spinner turbine from the shim system because it cannot be re-adjusted in the field with the necessary precision.

At installation of a new CryoProbe

Perform a **first spinning test**: Remove the shim system from the magnet (see **"Replacing the shim system (optional)" on page 24** in the *Installation Manual*) and put it in upright position onto a level surface. Remove the yellow spinner gas hose from the magnet top, put it into the yellow spinning gas inlet on top of the shim system (the other inlet is the sample lift), and switch on sample spinning. If the sample does not spin properly, the spinner turbine must be cleaned.

IMPORTANT: Do not remove the blue spinner turbine from the shim system because it cannot be re-adjusted in the field with the necessary precision.

The **second spinning test** involves the CryoProbe. Be extremely careful with the fragile tube of the CryoProbe! Put CryoProbe on a level surface with the shim system on top of it and repeat the first spinning test. If the sample doesn't spin properly, the axial alignment of the blue spinner stator does not match the vertical axis of the CryoProbe (the tolerances are very tight for sample spinning in a CryoProbe). This alignment was done at the factory. Since there are many degrees of freedom for this adjustment, it is virtually impossible to improve the alignment in the field and the shim system must be exchanged.

If another CryoProbe is available, test its spinning properties for comparison.

Indicator on CryoProbe sticks out

5.2.3

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

CryoController failure

5.2.4

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

The CryoController inside the CryoCooler cabinet has an overheat protection.

Leak in He supply

5.2.5

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

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

Cool-down problems

5.3

COOL DOWN command is ignored

5.3.1

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

5.3.2

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 the He Transferline had been twisted recently, the vacuum flange on the He Transferline may be closed properly, see **"Joining the CryoCoupler" on page 34**,
- if you hear a squeal sound that comes and goes, the turbo vacuum pump may not come to speed. Check its status display inside the CryoCooling Unit cabinet to the left.

General vacuum test

Press *COOL DOWN* and wait for diagnostic messages.

CryoProbe vacuum test

Enter the *CryoTool Service* mode and let the vacuum system run as if the system were cooling-down or in cold operation, i.e. V10 and V11 open, Sw2 *ON*, Sw3 *ON* and *ON SPEED*. The Vacuum Plug must have been drawn out of the CryoProbe with the help of the Vacuum Adapter. When the readings of *Vac CRP* and *Vac CU* are stable, write down their values. Then, use the Vacuum Adapter to push the Vacuum Plug into the CryoProbe. If *Vac CRP* drops sharply, there's a leak in the CryoProbe which must be repaired at the BRUKER factory.

Section test

Enter the *CryoTool Service* mode and let the vacuum system run as if the system were cooling-down or in cold operation, i.e. V10 and V11 open, Sw2 *ON*, Sw3 *ON* and *ON SPEED*. Use the Vacuum Adapter at the CryoProbe, V10, and V11 to isolate sections of the vacuum system one after another. Monitor the readings of *Vac CRP* and *Vac CU* which are located at opposite ends of the vacuum system, i.e. inside the CryoProbe and at the Coldhead, respectively.

He Compressor does not run**5.3.3**

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

Table 5.1. Troubleshooting the He Compressor

Symptom	Possible cause	Elimination
<i>MAIN POWER</i> and <i>DRIVE</i> remain <i>ON</i> when switched but the He Compressor does not start after He flushing is finished.	<ol style="list-style-type: none"> 1. No mains power. 2. Abnormal room temperature. 3. Incorrect phasing of mains. 4. DC control circuit malfunction. 	<ol style="list-style-type: none"> 1. Check mains wiring, fuses, and circuit breakers. 2. Maintain room temperature at 5° - 35°C. 3. Check phasing of input power. 4. Check DC 24 V circuit and fuses.
<i>MAIN POWER</i> remains <i>ON</i> but <i>DRIVE</i> flips back to <i>OFF</i> .	<ol style="list-style-type: none"> 1. Abnormal static He pressure. 	<ol style="list-style-type: none"> 1. Adjust He supply pressure.

Troubleshooting

Table 5.1. Troubleshooting the He Compressor (Forts.)

Symptom	Possible cause	Elimination
He Compressor stops after several minutes of operation and <i>DRIVE</i> goes <i>OFF</i> .	<ol style="list-style-type: none"> 1. Abnormal operating He pressure. 2. air-cooled only: Insufficient cooling of compressor operating room triggers thermal protection. 3. air-cooled only: Air flow for He Compressor is blocked. 4. air-cooled only: Air flow through the He Compressor unit is blocked by dirty fin. 5. air-cooled only: Fan does not operate. 6. water-cooled only: Insufficient or no water cooling triggers the thermal protection. 7. Inadequate voltage at AC mains source. 8. Excessive current of the He Compressor capsule. 	<ol style="list-style-type: none"> 1. Adjust He supply pressure or charge He as required. 2. Check room temperature and cooling system of compressor operating room. 3. Check the space around the He Compressor. 4. Clean the heat exchanger fin. 5. Check fuses (see "Fuses and reset buttons" on page 59), if OK then replace fan. 6. Confirm that cooling water is flowing at the proper flow rate. 7. Check mains voltage selector (European version P/N O00247 only), mains wiring, fuses, circuit breakers, and phasing. 8. Measure the current of the He Compressor. If a high and sustained motor current is measured, replace the He Compressor unit.
He Compressor has shut down but the switches remain <i>ON</i> .	<ol style="list-style-type: none"> 1. No mains power. 2. Abnormal operating He pressure. 3. Abnormal He pressure due to gas leak. 4. Abnormally high discharge He temperature due to high room temperature. 5. Abnormally high discharge He temperature due to failure of solenoid valve or high pressure switch. 6. Abnormally high discharge He temperature due to insufficient oil flow in He Compressor. 	<ol style="list-style-type: none"> 1. Check mains wiring, fuses, and circuit breakers, see "Fuses and reset buttons" on page 59. 2. Adjust He supply pressure or charge He as required. 3. Check He pressure, charge He, look for leak. 4. Check room temperature and cooling system of operating room. 5. Restart the unit and check the pressure. If it is abnormal, check solenoid valve or high pressure switch. 6. Look for an oil leak. If there is no leak, replace the He Compressor unit.

Charge He Compressor

Usually, the CryoProbe System is charged with He at the CryoCooling unit and not at the He Compressor. If there's too little pressure in the He Compressor, i.e. its gauge reads less than 17.5 bar, it is recharged automatically upon cool-down after the preparations have been finished.

Reduce pressure at He Compressor

The He pressure in operation is regulated by the CryoCooling unit in full automation and no adjustment should be necessary.

To reduce the He pressure at the He Compressor before starting, see its operation manual which is delivered with the CryoPlatform.

Periodic hiss is missing**5.3.4**

Symptom: Cool-down is started, the He Compressor runs but the characteristic periodic sound from the CryoCooling Unit is missing.

Possible cause: The plate inside the ColdHead is either in the wrong position or it is blocked. This may happen, when the pressure on the Return is higher than on the Supply due to interchanging the order of connecting the two Flexlines.

Corrective action: A higher pressure has to be built up on the supply line of the ColdHead. Drive at the He Compressor front must be Off.

step	action
1.1	Disconnect the <i>SUPPLY</i> and then the <i>RETURN Flexline</i> from the Cryo-Cooling Unit .
1.2	Close all valves. (Notice that V4 and V8 are normally open!)
1.3	Open V7 and V8 to relief the pressure of the <i>RETURN</i> .
1.4	Close V7 and V8
1.5	Adjust now the pressure of the He gas supply with the pressure regulator to 17 bar. If the pressure is too high open V5 and V9 and relieve it by opening V8 for a short time.
1.6	Close all valves
1.7	Now switch <i>ON</i> the <i>DRIVE</i> of the ColdHead (which is located on the back side of the He compressor) and open V7 and V8. Then open and close V6 several times for a few seconds. With this action the pressure on the <i>SUPPLY</i> will be higher than the pressure of the <i>RETURN</i> .
1.8	Repeat the last step up to five times. As soon as the characteristic hiss sound is heard, switch <i>OFF</i> the <i>DRIVE</i> of the ColdHead and close all valves and re-establish the original configuration. If the periodic hiss cannot be heard, the valve might be blocked and needs to be handled as described in the next steps.
1.9	Close all valves (V4 and V8 are normally open!). Connect the large DumpTool to the <i>SUPPLY</i> of the ColdHead . Open its relief valve completely!
1.10	Open V5, V7 and V9
1.11	Switch on the <i>ON</i> the <i>DRIVE</i> of the ColdHead (which is located on the back side of the He compressor)

step	action
1.12	Close and open V7 five times for a few seconds. Switch <i>OFF</i> the <i>DRIVE</i> of the ColdHead .
1.13	Close the relief valve of the large DumpTool completely and close all valves (V4 and V8 are normally open!).
1.14	Repeat steps 1.7 and 1.8.
1.15	If the characteristic hiss can still not be heard, then the ColdHead needs to be replaced.

Cool-down doesn't reach cold state

5.3.5

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

Did the message *Warm-up needed soon* appear recently? If yes, see **“Warm-up needed soon request” on page 63** in the *User Manual*.

Adjusting the He flow

IMPORTANT: He flow adjustments at the CryoCooling Unit are sometimes counter-intuitive but always time consuming. Do not change any setting without explicit advice from BRUKER.

When cooling down a CryoProbe System for the first time, the flow of cold He may not be adjusted in an optimum fashion for the given environment, e.g. if the CryoCooling Unit was tested at a different mains frequency and voltage settings.

The overall He flow is factory-set at the (warm) throttle valve R1. It is interesting to note that a higher flow does not necessarily result in a better cooling. Thus, there is an optimum position that is different from ‘completely open’.

Balancing is done at the cold (!) throttle valve V16 which is located inside the CryoCooling Unit below the big cylindrical vacuum chamber. It carries a 360° scale and is closed by turning to the right, i.e. towards increasing values. This valve must not be operated when the CryoProbe System is cold.

Example: the ‘Preamp’ temperature is too low and cannot be increased even by 100% Preamp-H6 heater power.

Quick’n’dirty fix: reduce the overall flow while the system is in cold operation by closing R3: Mark the actual position of R1. Turn R1 in steps of quarter-turns and allow at least 10 min for equilibration.

Permanent solution: Close V16 in steps of half-turns with R3 being in its original position. Since V16 must not be operated when cold, each adjustment involves a full warm-up/cool-down cycle. To obtain reproducible results, close V16 first completely, the open it by the desired number of turns. After each change, cool-down, and equilibration for ½ - 1 h, write down the resulting *Insert* and *Preamp* temperatures to assure that the process goes into the right direction.

How to open the CryoCooling Unit

5.4

There are no user-serviceable parts inside the CryoCooling Unit.

WARNING: Put on protective goggles before opening the CryoCooling Unit cabinet. Be careful with the panels, there might have sharp edges. If you need to operate the CryoCooling Unit under pressure while open, put on an ear protection: in case of an overpressure, the release valves will act with an extremely loud bang!

The housing of the CryoCooling Unit can be almost completely removed. First, switch *OFF* the mains at the front. Remove the white plastic covers from the panels you want to remove. Unlock the 6 screws around the top with an 8 mm Allen key by a half turn counter-clockwise. **Two persons are needed to lift the heavy panels!** Pull the grounding cables away from the top and the walls. Now the back can be unlocked. Disconnect the backside ventilators mains by pressing on their connector's sides while pulling. Remove the side walls at last if necessary.

WARNING: When reassembling, make sure to connect all the grounding cables to the panels and the fan supply cable to the back panel. Verify the fan operation when the CryoCooling Unit is powered-up again.

Refill He

5.5

The *SUPPLY PRESSURE* gauge on the He Compressor front must not read significantly less than 16 bar (= 1.6 MPa or 16.3 kgf/cm²) at the time a cool-down is started, otherwise the cool-down will fail. If this cool-down failure just occurred, continue directly with step **f.3** in **Table 5.2.** below.

When a substantial amount of He is lost continuously during operation, the CryoController will give up automatic refilling and warm-up the system. Wait until *WARM* and *UNPLUG* are lit before refilling He.

NOTE: Do not try to fill the He Compressor directly from the He steel-cylinder (i.e. at its backside *HELIUM GAS CHARGE* port), it should be charged via the CryoCooling Unit.

Table 5.2. Refill He

step	action
f.1	To prevent errors due to other causes, the CryoProbe System should be ready for a cool-down in all other respects. Check if the 1) cooling water supply is active (if applicable); 2) He steel-cylinder is connected and open. Its primary pressure must be larger than 40 bar and its secondary pressure stable at 22-25 bar; 3) CryoProbe is properly installed and connected; 4) CryoCooling Unit is <i>ON</i> while <i>WARM</i> and <i>unplug</i> indicate that the unit is ready; 5) He Compressor <i>MAIN POWER SW</i> at its backside and <i>DRIVE</i> at its front are <i>ON</i> .
f.2	Start <i>COOL DOWN</i> and wait until it stops with an <i>ERROR</i> .
f.3	Enter the password-protected <i>Service</i> mode of the <i>CryoTool</i> and click <i>debug HALT - Yes</i> .
f.4	Close the He Regulator.
f.5	In the <i>CryoTool</i> , close <i>V1</i> , <i>V2</i> , <i>V3</i> , <i>V4</i> and <i>V8</i> .
f.6	Open <i>V6</i> and <i>V7</i> .
f.7	Open <i>V5</i> .
f.8	Now the timing is important: Open the He Regulator slowly in small increments such that the <i>HE-Supp.</i> and the <i>HE-Ret.</i> readings in the <i>CryoTool</i> approach 17.5 ± 0.3 bar slowly. As soon as one of them shows 17.5 bar, close <i>V5</i> immediately. NOTE: The gauge on the He Regulator is of less concern but it must not exceed 25 bar at any time.
f.9	Exit the <i>Service</i> mode.
f.10	Switch the CryoCooling Unit at its front <i>OFF</i> , then <i>ON</i> . The system now ready for a normal <i>COOL DOWN</i> .

Relocation of a CryoPlatform

6

Relocation

6.1

This section applies to moving a CryoProbe System to either a storage room or to another NMR spectrometer. If storage in a pressureless state is required (not recommended!), see **"Pressure release" on page 70**.

For disassembling the CryoProbe System, use the reverse order of the installation sequence **"Initial setup" on page 17**. Consider also the *CryoProbe Site Planning Guide*.

Before moving the CryoProbe System to a different magnet, make sure that the new magnet qualifies (clearance between shim system and floor, blue spinner turbine stator etc.).

CryoProbe System storage

6.2

The CryoProbe System must be stored in a clean, dry, and non-corrosive atmosphere. Minimum requirements are: -20° to + 50°C, humidity max. 95% (non condensing).

Put the Protection Cap on the CryoProbe sample cavity and make sure that the Vacuum Plug is in place at the bottom. Close the CryoCouplers on CryoProbe and He Transferline tightly with their special protective caps. Wrap the whole CryoProbe (or at least its tube) in airtight plastic film and stow it in its case. There is no preferred storage orientation for the CryoProbe case.

Use individual airtight plastic bags for all small parts like Tuning Adapter, Vacuum Adapter etc.

CAUTION: Any contamination can have severe effects on shimming, background signal, vacuum insulation etc.

Disposal

6.3

Disassembly and disposal of a CryoPlatform should be carried out by technically trained personnel only.

Relocation of a CryoPlatform

Pressure release

6.3.1

To permanently release the gas pressure from the CryoCooling Unit, shut down the whole system but keep the Flexlines and the He Compressor connected. Slowly unlock the pipe connectors marked in **Figure 6.1**, and let the gas escape. Leave the nuts unlocked but keep the connectors on their threads.

Figure 6.1. Releasing the gas from the CryoCooling Unit



Toxic materials

6.3.2

Lead insulation in CryoCooling Unit

The panels of the CryoCooling Unit cabinet contain a lead-enforced polymer foam for maximum sound insulation. Remove the lead/foam insulation. Recycle or dispose it according to the local regulations for lead-containing materials.

Indium seals

There are two indium layers inside the CryoCooling Unit and two indium seals inside the CryoProbe.

Inside the CryoCooling Unit, open the big vacuum chamber. The Coldhead is mounted inside of an arrangement of helical heat-exchanger pipes. There are indium layers in the two contact faces between Coldhead and helix. Unlock the screws and remove the indium.

At the CryoProbe, remove the semicircular bottom plate to gain access to another large plate in the shape of a ring. Lift the plate. The two indium o-rings make the seal between the ring-shaped plate and the body.

Indium must be recycled or disposed according to local regulations.

BRUKER contact

7

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

7.1

CryoProbe information head offices:

BRUKER AG
Probe Department
Industriestrasse 26
CH-8117 Fällanden
Switzerland

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

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

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

CryoProbe service

7.2

CryoProbe service head offices:

BRUKER AG
Service Department
Industriestrasse 26
CH-8117 Fällanden
Switzerland

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

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

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

Check lists

A

Site preparation checks

1.1

Before an installation date with BRUKER is fixed, the customer has to make sure that the following prerequisites are fulfilled in the NMR laboratory.

- Can the CryoProbe System be installed according to the site planning that was agreed on with BRUKER or did something change in the meantime? If yes, contact BRUKER immediately!
- Are all supplies available (mains, pneumatic gas, cooling water...)?
- Do all supplies carry the required connectors?
- Do other devices in the laboratory depend on the same supplies as the CryoProbe - in particular: pneumatic gas and electricity? Is there a need for gas buffers, check valves, or a UPS? Can the other laboratory equipment continue its operation during the CryoProbe installation or does it have to be switched off?
- Is the path for all cables and tubes associated with the CryoProbe System prepared (space, cable channels, holes...)?
- Is the space below the magnet between shim system bottom plate and floor cleared?
- Is the acoustic insulation for the He Compressor and the optional water chiller ready (adjacent room or cabinet)?
- Is there enough space for delivery, unpacking, and installation? Is a hoist available for unpacking the CryoCooling Unit?
- Will a second person be occasionally available to lift the heavy CryoProbe into the magnet?
- Is a recent set of NMR reference data available that can document the spectrometer performance, e.g. in terms of lineshape, sensitivity, stability, spurious signals etc.?

Preparations for the installation visit

1.2

When planning and preparing an actual CryoProbe installation, the Bruker office must consider the following points.

- Have all CryoProbe System parts been shipped?
- Are all required tools at hand?
- Are the latest versions of the CryoProbe System documents and software available, e.g. *Installation*, *Site Planning* and *User* manuals, *Firmware* and *CryoTool*?

- Is there a definite drawing of the CryoProbe System layout for the customer's NMR lab?
- Will all connectors on the Bruker parts fit the supplies in the customers laboratory (mains, gas, water, ...)? Mind the different national standards.
- Bring the required documents (Acceptance, Service Report, installation protocol, expenses...)
- Prepare passwords etc. for optional remote access.
- Bring a selection of in-line attenuators for use as safety attenuators.
- If a sample changer has to be moved: bring tools and manuals.
- If the magnet has to be moved or lifted: bring tools.
- Double-check if the customer shim-system is equipped with a blue spinning stator. If in doubt, prepare an exchange shim system.
- Is a receiver or ADC upgrade requested by the customer?

User training

1.3

The following topics should be addressed to familiarize the users - in particular the NMR superuser - with a new CryoProbe System:

- System overview.
- Safety (pressurized cryogenic gas cycle, mains failure, sample safety, wearing protective goggles and gloves, etc.).
- User documentation.
- Differences to a conventional NMR probe.
- Standard procedures like mounting the CryoProbe, cool-down, wobbling, shimming etc.
- Experimental parameters like RF power, receiver gain, operating temperature, sample tubes etc.
- Heating due to decoupling.
- Sample temperature control.
- Preamplifier selection (*crpon* etc.), CryoTool, Firmware update.
- Safety attenuators (optional).
- Cabling, HPPR CRP internal wiring, external RF filters, re-configuration for conventional probes.
- Handling and cleaning the CryoProbe.
- O-ring handling.
- Monitoring the system, e.g. logbook, system messages, blink codes etc.
- User maintenance (He refill, He steel-cylinder exchange, etc.) and periodic service by BRUKER.
- BRUKER support and service: addresses, logfile, fault report, repair declaration, description of circumstances etc.
- Shutdown and storage.

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 Preparation Manual (P/N Z31553)

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

CryoProbe data sheets

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

He Compressor technical manual

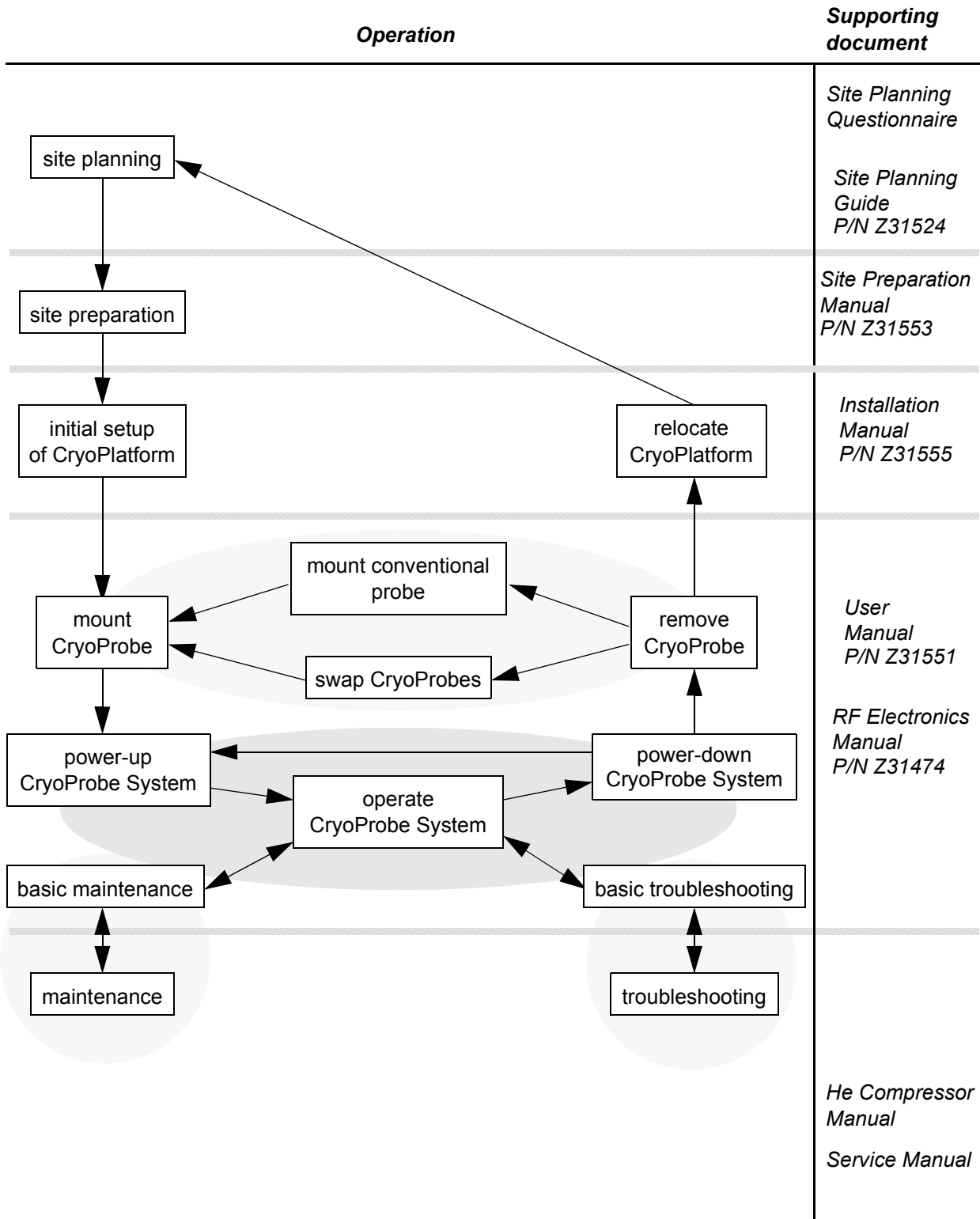
The operation manual is delivered with the He Compressor.

CRP RF Electronics Technical Manual (P/N Z31474)

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

Related documents

Figure A.1. When to use which CryoProbe document



Conversion of metric units

B

$$1 \text{ bar} \equiv 0.1 \text{ MPa}$$

$$1 \text{ bar} \approx 14.5 \text{ psi}$$

$$1 \text{ bar} \approx 1.02 \text{ kgf/cm}^2$$

$$1 \text{ kg} \approx 2.2 \text{ lb}$$

$$1 \text{ mm} \approx 0.04 \text{ inch}$$

$$1 \text{ m} \approx 3.28 \text{ feet}$$

$$1 \text{ Nm} \approx 8.85 \text{ lbf-inch}$$

$$1 \text{ L (liter)} \approx 0.264 \text{ gallon (U.S.)}$$

$$1 \text{ L (liter)} \approx 0.220 \text{ gallon (Brit.)}$$

$$1 \text{ kWh} \approx 3.6 \text{ MJ}$$

$$1 \text{ kWh} \approx 3412 \text{ btu}$$

$$1 \text{ mT} \equiv 10 \text{ Gauss}$$

°C to °F:

$$T_{\text{°F}} = (T_{\text{°C}} \times 1.8) + 32$$

$$1 \text{ Pa} \equiv 0.01 \text{ mbar}$$

$$1 \text{ psi} \equiv 68.95 \text{ mbar}$$

$$1 \text{ kgf/cm}^2 \approx 0.98 \text{ bar}$$

$$1 \text{ lb} \equiv 0.4536 \text{ kg}$$

$$1 \text{ inch} \equiv 25.4 \text{ mm}$$

$$1 \text{ foot} \equiv 0.3048 \text{ m}$$

$$1 \text{ lbf-inch} \approx 0.113 \text{ Nm}$$

$$1 \text{ gallon (U.S.)} \approx 3.79 \text{ L}$$

$$1 \text{ gallon (Brit.)} \approx 4.55 \text{ L}$$

$$1 \text{ MJ} \approx 0.278 \text{ kWh}$$

$$1 \text{ btu} \approx 0.293 \text{ Wh}$$

$$1 \text{ Gauss} \equiv 0.1 \text{ mT}$$

°F to °C:

$$T_{\text{°C}} = (T_{\text{°F}} - 32) / 1.8$$

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

Glossary

C

Adsorber

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

BBIS

BRUKER Board Information System

Coldhead

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

CryoController

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

CryoCooler

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

CryoCooling Unit

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

CryoCoupler

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

CryoPlatform

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

CryoPreamp

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

CryoProbe

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

CryoProbe RF Unit

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

CryoProbe System

A CryoProbe and all components necessary for its operation.

CryoTool

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

Dump Tool

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

Flexlines

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

Gradient Filter Box

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

He

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

He Compressor

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

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

He Hose

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

He Regulator

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

He steel-cylinder

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

He Transferline

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

HPPR CRP

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

HPPRtool

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

Magnet stand pillar braces

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

Mounting Hardware

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

PIC

Probe Identification and Control system 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 quality factor Q is a measure of the efficiency of reactive devices such as inductors, capacitors, or resonant circuits.

Radiator

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

RF

Radio frequency

Transferline Support

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

Tuning Adapter

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

Tuning Tool

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

UniTool

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

UPS

Uninterruptable Power Supply, a kind of battery that compensates for fluctuations and interruptions in the mains.

Vacuum Adapter

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

Vacuum Plug

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

Vacuum bellows

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

Vacuum system

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

VT gas

Usually nitrogen gas or dry air at a controlled variable temperature that flows through a probe to heat or cool the sample. Its function must not be confused with

the 'pneumatic gas' used for operating valves inside the CryoCooling Unit or with the helium gas circulated through the CryoProbe for cryogenic cooling.

VT Interface Box

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

VT unit

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

Water chiller

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

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

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