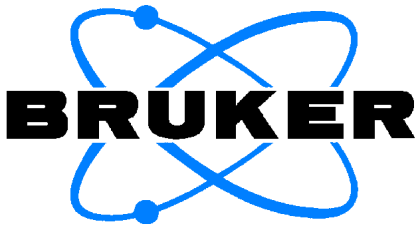

CryoProbe System

Installation

Version 2.0

BRUKER

This manual was written by

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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.

Personnel safety *1.4*

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

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

If in doubt: Put on goggles and protective gloves!

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

Inherent safety

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

CE certification

CryoProbe, CryoCooling Unit and He Compressor are CE certified.

Technically qualified personnel only

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

No user-serviceable parts inside

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

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

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

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

Pressurized cold helium gas cycle

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

The helium gas volume inside the cryogenic cooling cycle is small and presents virtually no danger of suffocation. However, the He steel-cylinder contains a substantial gas volume, note the warning below (***"Pressurized helium gas supply" on page 8***).

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.

Introduction 2

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

How to use this manual 2.1

This *CryoProbe System Site Planning Guide* will help you to find out if an NMR laboratory qualifies as a site for a CryoProbe System, which preparations are necessary for a smooth installation and operation, and which options should be considered before placing an order.

Use the appropriate ***"Check lists" on page 63*** 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 5*** .

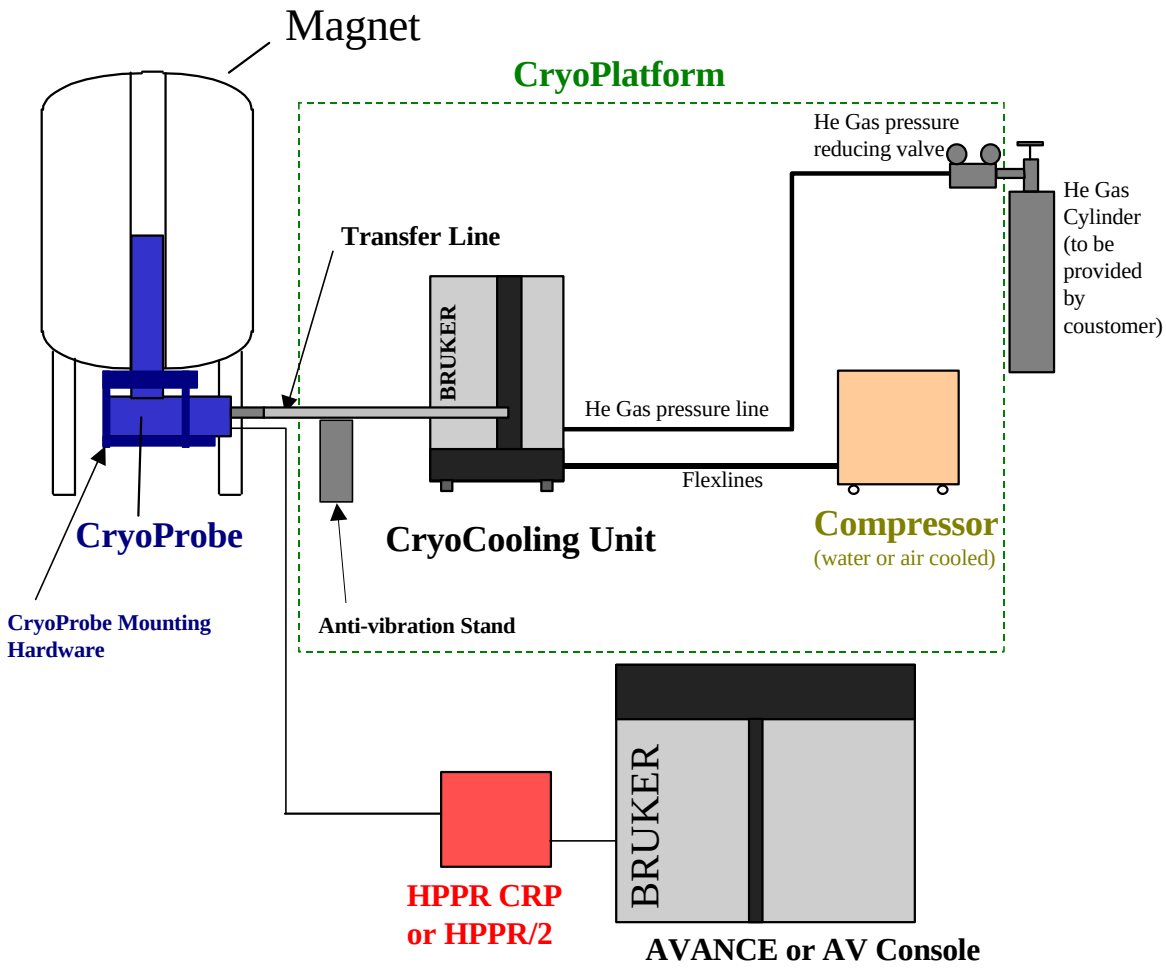
Further information can be found in the manuals listed in ***"Related documents" on page 67***.

CryoProbe System overview 2.2

A CryoProbe System consists of several subunits: CryoProbe, CryoPlatform, cryo-compatible HPPR CRP or HPPR/2, and the He steel-cylinder. (***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

SMALL CAPS ITALIC	setting of a hardware switch or button
`Courier small`	contents of a file
`Courier small italic`	system response
`Courier`	file or directory name
`Courier bold`	Unix™ or Windows™ NT keyboard command
`Courier italic bold`	BRUKER NMR Suite keyboard command
Times bold	Unix or Windows NT object clicked with the mouse
Times italic bold	BRUKER NMR Suite object clicked with the mouse
Times italic	host name, user name etc.
< >	place holder

Initial setup 3

Before delivery 3.1

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 63*** for the NMR laboratory.

The checklist ***"Preparations for the installation visit" on page 63*** 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!

Transport 3.2

The CryoProbe System is shipped in two steps: first a *Site Preparation Set* containing the He Compressor some tubes which must be installed by the customer in the NMR lab to provide the infrastructure for the CryoProbe System. Included with the first step are the CryoCooling Unit and HPPR CRP or HPPR/2 if not al-

ready on site. After the site preparation work has been completed and installed the CryoProbe is delivered in a second step.

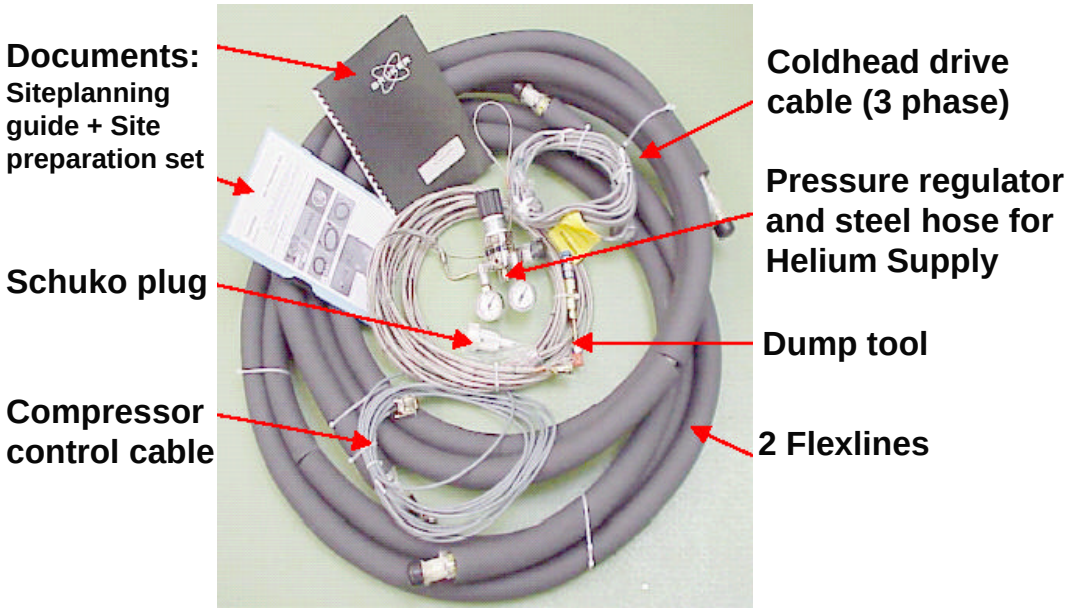
List of the Crates *3.2.1*

1.a **cardboard box** (water-cooled He Compressor)
or
1.b **cardboard box** (air-cooled indoor He Compressor)
or
1.c. **two cardboard boxes**: (air-cooled outdoor He Compressor)

A power cable with a length of 4m and with four wires (three phases and one ground) is delivered with the He Compressor. The water-cooled version has included two quick connectors for the chilling water hose.

2. **cardboard box:** (Flexlines and cables for He Compressor)

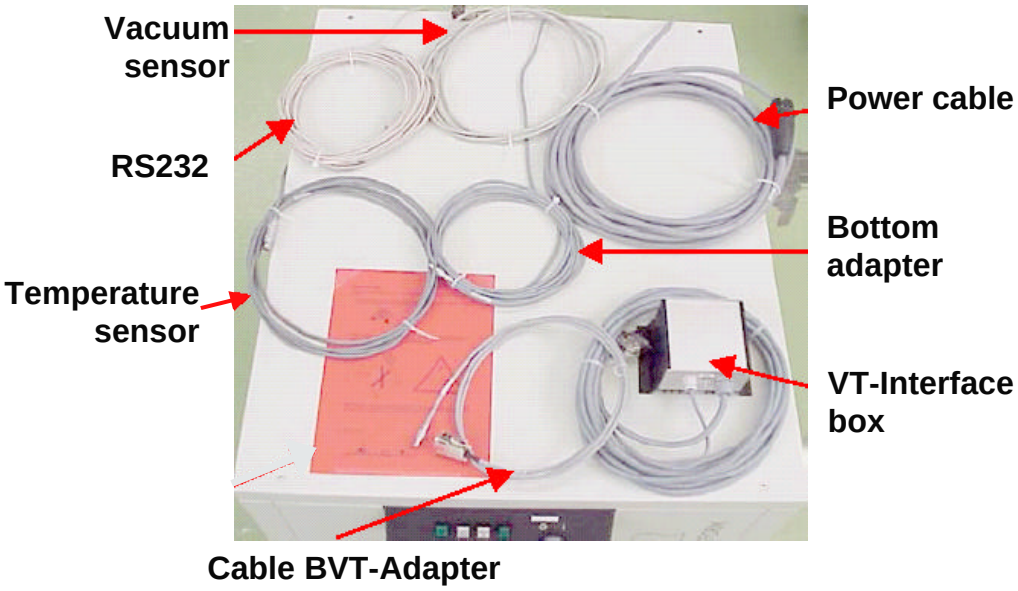
Figure 3.1. Flexlines and cables for He Compressor



3. **cardboard box:** outdoor Flexlines, one power cable, two control cables. (only for outdoor He Compressor)

4. **wooden crate** (CryoCooling Unit (400kg))

Figure 3.2. CryoCooling Unit



5. **wooden crate**: (Transferline support)

Figure 3.3. Transferline support

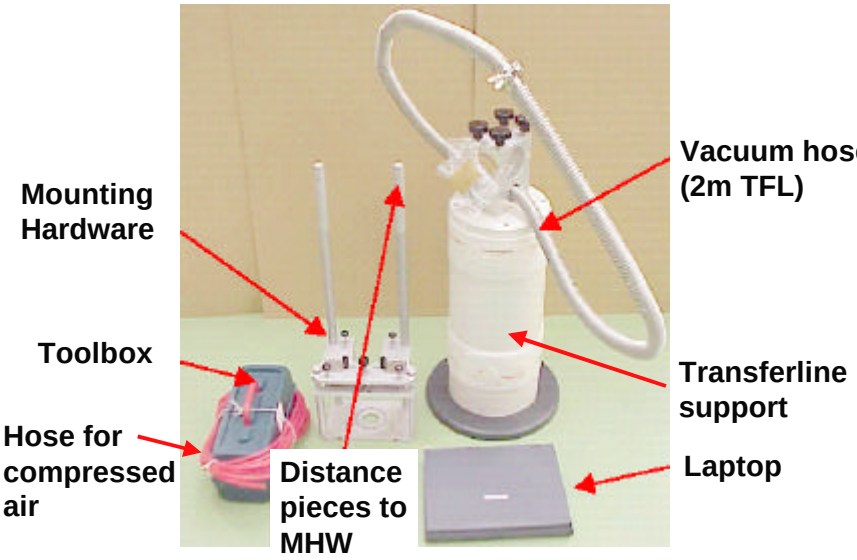


Table 3.1. Content of the Toolbox

	Tool	Where to use it
1	Titanium wrench 36mm	CryoCoupler
10	O-rings 7.10x1.60 viton	CryoCoupler
2	wrench 5/8” + 3/4”	Helium steel hose

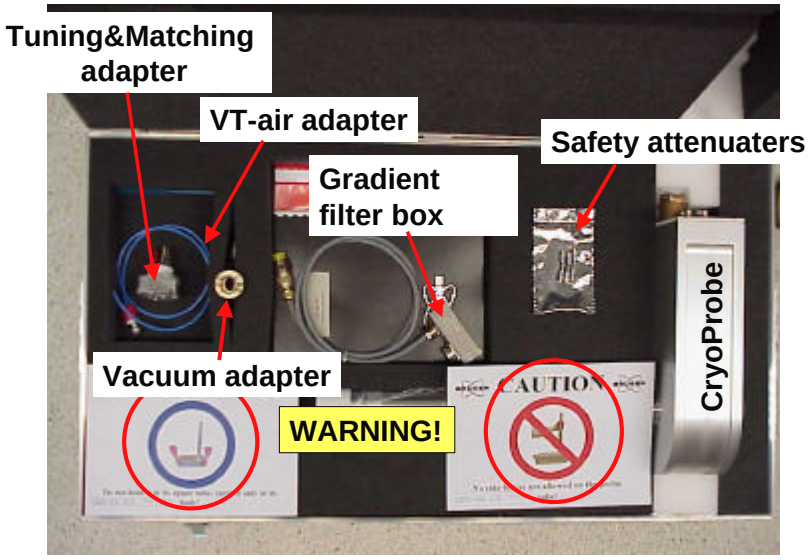
Table 3.1. Content of the Toolbox

	Tool	Where to use it
1	wrench 15/16”+ 1’’	Flexlines
1	wrench 1 3/16” + 1 5/16”	Flexlines, He-Regulator
4	screws M5x12	Mounting Hardware
1	Legris pneumatic T-piece 8 mm	compressed air
10m	Pneumatic hose 8/5.5 mm	compressed air
	SNOOP liquid	find a helium leak
	vacuum grease	o-rings, vacuum adapter
10	fuses 5x20mm 50 mA T	CryoCooling Unit

6. **cardboard box** 27 kg: HPPR CRP (optional)

7. **cardboard box**
pallet
flightcase 30 kg
i.e. in total 40 kg: CryoProbe

Figure 3.4. CryoProbe



+ additional items in cardboard boxes (e.g. pillar braces for the magnet, HPPR/2 modules, filters and cables, shimsystem with a blue stator turbine,...)

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..

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 63***.

Before you unpack *3.4*

REMARK: Since the customer is asked to connect the He Compressor prelimery to the installation visit, the crates containing the He Compressor and the Flexlines should already be unpacked.

Ensure safety

Read the instructions for ***"Safety" on page 5*** 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?

Check for completness

Are all wooden crates and cardboard boxes there according to ***"List of the Crates" on page 14*** ?

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!

Unpacking *3.5*

Use the shipping and packing lists and see also ***"List of the Crates" on page 14*** 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 17***. 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.

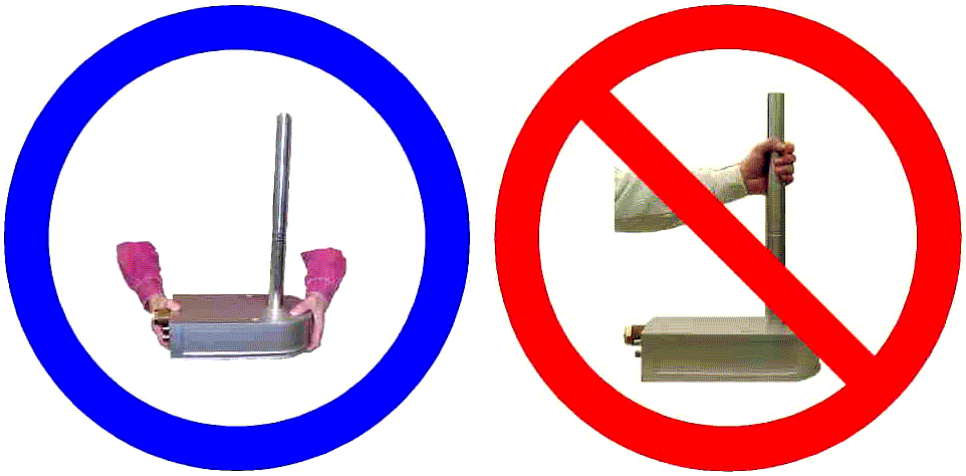
CryoProbe

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 3.5. CryoProbe handling



If only an additional CryoProbe is to be installed on a working CryoPlatform, continue with ***"Handling" on page 31***.

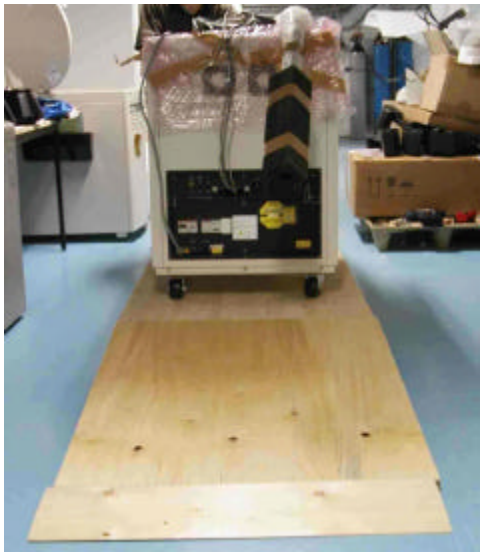
CryoCooling Unit/2

Disassemble the crate and remove the transport fixtures. The CryoCooling Unit weights ~400 kg. It is most convenient to roll it down the pallet using one of the side walls as a ramp. It can also be lifted with a fork-lift truck or with a hoist. Remove the panels of the CryoCooling Unit when lifting it with a hoist.

Figure 3.6. How to unpack the CryoCooling Unit/2

Lift first the fixation stand to remove all obstructing material. Then lower the stand completely before rolling the CryoCooling Unit down from the pallet.

Figure 3.7. Roll down the CryoCooling Unit/2 from the pallet



CryoCooling Unit/1

The CryoCooling Unit/1 has now wheels It can either be lifted of the pallet with a fork-lift truck or with a hoist. There are four eyes (for hooks or ropes) on the inner frame of the CryoCooling Unit.

IMPORTANT: Take special care of the He Transferline when unpacking and moving the CryoCooling Unit. Avoid tight bending! If the bending radius becomes too small, 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.

Start to evacuate the Coldbox *3.6*

As soon as the CryoCooling Unit has been unpacked, it should be evacuated. It is recommended to start this procedure as soon as possible and to let it last over night. The Coldbox has a large volume and a very large surface since insulating material made of many layers is wrapped around the cold parts. Humidity sticking on this large surface will require much time to be evauated.

Table 3.2. Evacuate the Coldbox.

setp	action
u.1	Position the CryoCooling Unit in the laboratory as agreed on during site planning.
u.2	**Remove all panels** from the CryoCooling Unit.

Table 3.2. Evacuate the Coldbox.

setp	action
u.3	Plug-in the CryoCooling Unit **mains**. **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 *OFF*. **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. **CAUTION:** Before powering-up The CryoCooling Unit 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
u.4	Attach **pneumatic gas** of at least the minimum specified pressure to the *Compressed air supply 4.5 - 8 bar* to 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.5	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.6	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 ***Service*** mode. They can easily lead to erroneous clicks with fatal results when operating e.g. individual valves.
u.7	Connect the CryoController's **RS232** output (located inside the CryoCooling Unit) to *COM1* port of the laptop (labelled *IOIOI* on its back) with the 9-pin computer cable Z14126. **IMPORTANT:** The CryoController's RS232 interface must not be used for other purposes!
u.8	Boot the laptop and login as **`cryouser`** (no password required).

Table 3.2. Evacuate the Coldbox.

setp	action
u.9	Turn the rotary main switch at the CryoCooling Unit front ***ON***. The Cryo-Controller will boot automatically. After a few moments the **green** *WARM* and the **white** *UNPLUG* **must be on**. Since not all sensors are connected jet, also the **red** *ERROR* will light up. **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 *REMOTE SWITCH* socket which short-circuits two pins.
u.10	Start the ***CryoTool*** on the laptop by either clicking its desktop icon or **Start - Bruker - CryoTool - CryoTool**. Select ***COM1***, click ***Service*** and enter the password to bring up the full interface and ***Log*** to see the system messages. Since not all sensors are connected jet, error messages will keep popping up. Just ignor them. **NOTE:** If the CryoTool is started before the CryoCooling Unit is switched *ON*, it will go to off-line mode because it sees no response at its *COM1* port.
u.11	Connect the vacuum cable (Z13846) to the separate vacuum sensor (Z48556) of the service kit (see ***Service Manual***). The vacuum sensors are only working, if both senors are connected due to the serial wiring of the heating filament.
u.12	In the Service Window of ***CryoTool*** observe the reading of Sensor 14. If the reading < 0.5 mV then start the membrane pump (sw2) and open V12. Wait until the reading becomes > 0.5 mV.
u.13	If the reading > 0.5 mV then start both pumps (sw2 and sw3) and wait until the turbo pump is on speed.
u.14	Open V12 and wait until the reading of Sensor 14 gets stable and is not increasing anymore. The reading for high vacuum is about 6 - 8 mV. Use the Graph in ***CryoTool*** to visualize the temporal evolution of the vacuum sensor reading. It may take several hours to get a high and stable vacuum. In the mean time the rest of the installation can be accomplished.

Test the He Compressor *3.7*

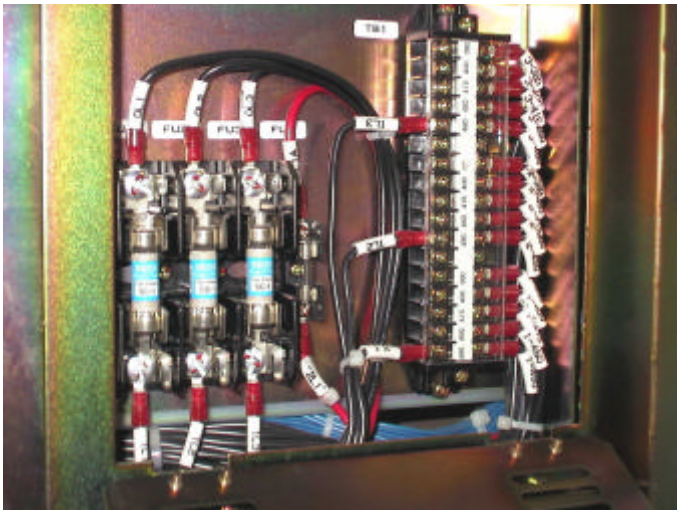
NOTE: Since the customer is required to have unpacked and plugged in the He Compressor, it should already be installed and connected.

An installation engineer is normally not allowed to manipulate the electrical mains of the He Compressor. Therefore is recommended to test it in the beginning of the installation since it may take some time to organise an electrician.

Check the SUPPLY PRESSURE gauge on the He Compressor front. It should indicate at least 0.1 bar (i.e. 0.01 MPa ≈ 0.1 kgf/cm^2). If the pressure gauge reads plain 0 bar, contact BRUKER: this is a major problem and a lengthy gas purifying cycle is required.

400V He Compressor only: Make sure the mains is not connected and set voltage selector according to the available mains voltage (380, 400, 415, 460 or 480V). The selector is either a switch or wire array according to. See also the He Compressor operation manual which is delivered with the CryoPlatform.

Figure 3.8. 400V Compressor voltage selector



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

Figure 3.9. Switches of the water-cooled He Compressor

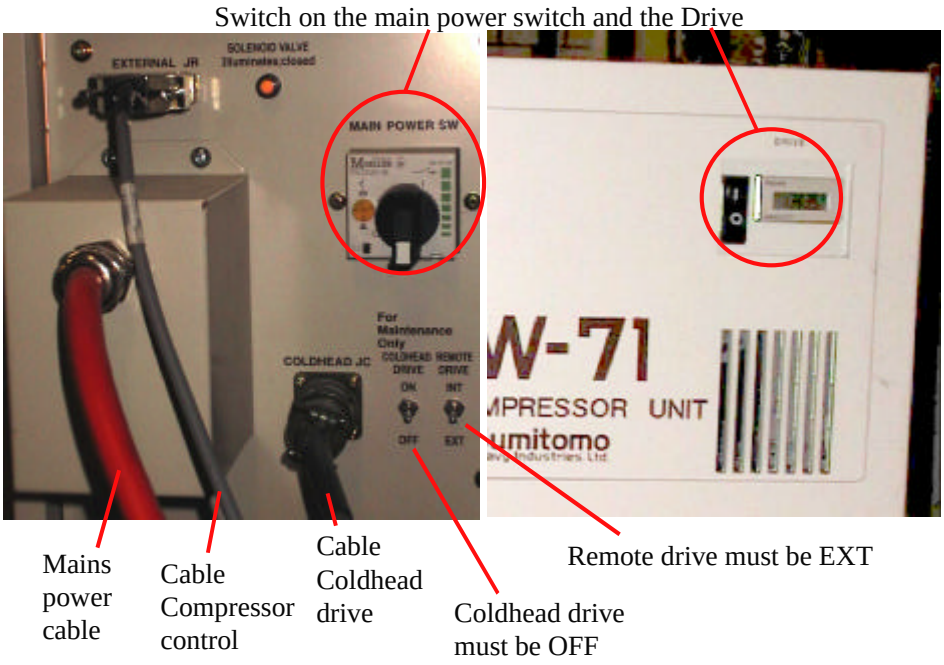


Table 3.3. Function test

setp	action
u.1	Make sure the mains is connected. **NOTE:** The SUPPLY PRESSURE gauge on the He Compressor front must read at least 1.5bar. Otherwise the He Compressor will not start at all, since it has a protective switch preventing it to run at a too low pressure. In such a case the He Compressor must be refilled according to ***"Refill the He Compressor (if necessary)" on page 50***.
u.2	Verify on the backside that the *COLDHEAD DRIVE* is *OFF* and *REMOTE DRIVE* in *EXT* position (see ***"Figure 3.9."***). **CAUTION:** If *COLDHEAD DRIVE* and *REMOTE DRIVE* are in the wrong positions, the Coldhead inside the CryoCooling Unit can be damaged.
u.3	Switch the **He Compressor** *MAIN POWER SW* at its backside ***ON***, then ***DRIVE*** at its front ***ON*** (see ***"Figure 3.9."***). The unit will go into **stand-by** mode.
u.4	Set the *REMOTE DRIVE* from the *EXT* position to the *INT* position for a couple of seconds. The Compressor should start immediately. **REMARK:** For the outdoor Compressor this test can only be done when the indoor and outdoor unit are connected with one another. This test will therefore be done later on. Since the switch is located in the indoor unit wich is normaly distant from the outdoor unit, the start is therefore not audible. The indoor unit has a lamp labled 'OPERATION' which will be lit when the Compressor is started.
u.5	Set the *REMOTE DRIVE* back from the *INT* position to the *EXT* position.
u.6	If the He Compressor did not start, then the phasing is most probably not correct. The Compressor has a built-in protection against a wrong orientation of rotation. Disconnect the mains power cable and interchange two phases with each other. Repeat the steps u.1 to u.5

Positioning the units *3.8*

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 3.4. Position the units

step	action
p.1	**Place all units** in the laboratory as agreed on during site planning: CryoCooling Unit with He Transferline, Transferline Support and He Compressor. Install the He Compressor on a level surface. Two of its four casters should be locked. **Provide extra space around the units** as required for connections, ventilation, and service (details are given in the *CryoProbe System Site Planning Guide*).
p.2	**NOTE:** Orientation of the Transferline Support: the screw fixture of the vacuum tube must face the CryoCooling Unit. Connect the shorter **vacuum tube** from the Transferline Support to the vacuum port at the CryoCooling Unit front, right below the He Transferline outlet. **NOTE:** On the earliest CryoCooling Units, the vacuum port is not accessible from outside. Open the cabinet and connect the *longer* (!) vacuum tube to the port inside.
p.3	**Lock the He Transferline** to the Transferline Support with the four big screws.
p.4	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.5	Put the dark-grey **basement cover** to the CryoCooling Unit front bottom.

BCU05 cooling test (optional) *3.9*

If a gas cooler, e.g. a BCU05, shall be used with the blue non-isolated VT hose between its outlet and the CryoProbe, its output temperature must be tested and adjusted. 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 3.5. 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 *3.10*

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 *3.10.1*

A spatial channel of at least 195 × 578 / 628 / 692 or 853 mm [width × height] is needed from the magnet front to insert a 500 / 600 / 700 or 800 - 900 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.

Cranked magnet pillar braces (optional) *3.10.2*

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 3.6. Mount the cranked magnet pillar braces (optional)

step	action
s.1	**Check the gap** between the lower magnet pillar brace and the floor: it must be at least 95 mm. 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 *Mechanical Levelling of the Anti Vibration Stand* in the magnet manual.
s.2	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.
s.3	**Switch *OFF*** the magnet's air suspension.
s.4	**WARNING:** Do not remove a brace if the magnet is still suspended or if the 9 fixing screws are not in place! **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.
s.5	Replace the **second brace** only after the first brace has been tightened.
s.6	Remove the **9 fixing screws** from the magnet stand.
s.7	Do **not** switch *ON* the magnet's air suspension yet but wait until the CryoProbe is mounted.

Blue spinning stator *3.10.3*

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.

Replacing the shim system (optional) *3.10.4*

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 3.7. 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	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. **CAUTION:** Do not touch the Allen key screws in the magnet's flat bottom flange! **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 ***"Attach the Mounting Hardware" on page 29***.
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 *ON* 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 ***"Attach the Mounting Hardware" on page 29***.

Attach the Mounting Hardware *3.10.5*

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 (Z46554, Z46832, Z47166, Z47168 or Z70720)

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) (inside the Toolbox delivered with the CryoCooling Unit)

1 3mm 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

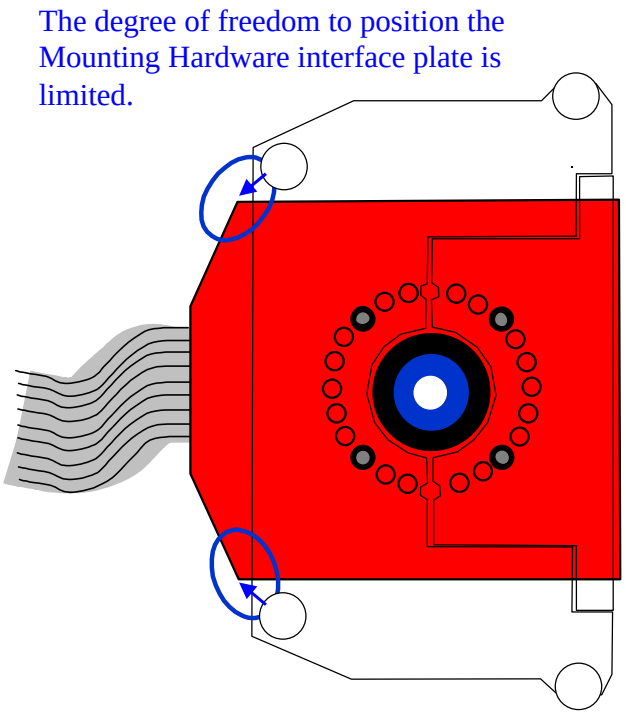
4 M5×20 Allen key screws, non-magnetic (11127 each) instead of M5×12

Table 3.8. Attach the Mounting Hardware

step	action
h.1	**Switch *OFF*** the magnet's air suspension.
h.2	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. **IMPORTANT:** If the shim upper part is not tigthly fixed a risk will persist of moving the entire shim sytem upwards when mounting the CryoProbe. **Mark the exact position** of the shim system at the magnet top with scratches or a permanent pen.
h.3	Remove the red shim system fixture **clamp at the magnet bottom**. **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.

h.4	**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 *CryoProbe System Site Planning Guide* for a 1:1 drawing of the Mounting Hardware interface plate (the interface plate is the part that touches the magnet). 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!
h.5	Check if there are **four empty threads** in the magnet bore bottom flange at a diameter of 76 mm.
h.6	**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. 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.
h.7	Take the CryoProbe **support plate** out of the Mounting Hardware.
h.8	Remove the **clamp part** that is integrated in the interface plate.
h.9	**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. **NOTE:** There are two cases in which four longer non-magnetic Allen key screws (M5×20) must be used instead of M5×12: a) 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. b) If screws had to be removed from the magnet bottom flange to provide four empty threads (see step ***h.5***).
h.10	**Insert the clamp part**, and fix it to the interface plate but do not tighten these screws. The function of the clamp part is only to guide the shim system but not to fix it. Make sure that it can still move up and down. The verical position of the shim system is still defined by the upper red clamp on the top of the magnet. This must be tighten firmly to avoid a shift of the shim system by the insertion of the CryoProbe. Put the remaining two screws in place, and tie all four screw in a balanced fashion, which attach the interface plate and the clamp part to the magnet. **IMPORTANT:** Make sure that the shim system tube is not tilted inside the magnet bore. Center it properly to avoid shimming problems. If the shim system had been rotated temporarily in step ***h.6***, bring the shim system back to its original orientation.
h.11	Do **not** switch *ON* the magnet's air suspension now but wait until the CryoProbe is mounted.

Figure 3.10. Mounting Hardware interface plate on magnet bottom



Handling *3.11*

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"*** in the *User Manual* 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 3.11. Never hold a CryoProbe at its tube!

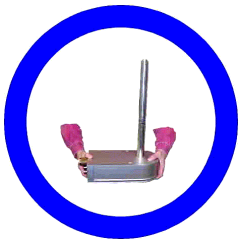


Figure 3.12. Carry a CryoProbe only at its body!

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

Mounting *3.12*

See the corresponding section in the *User Manual*.

Sample spinning test *3.13*

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.

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

Set the VT gas flow rate to 670 L/h and make sure that the VT hose connections are leak-tight. 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*.

If spinning does not work, see the *Service Manual*.

Joining the CryoCoupler *3.14*

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 3.9. Join the CryoCoupler

step	action
j.1	**Detach the He Transferline** from the Transferline Support but let it rest on the column.
j.2	**CryoCooling Unit/2:** Do the necessary adjustments only at the vacuum flange next to the Coldbox inside the front side panel of the CryoCooling Unit. **CryoCooling Unit/1:** Do the necessary adjustments only at the second vacuum flange when seen from the CryoProbe end. Unlock all six screws around the **vacuum flange** which is labelled ***Service only***, just a little. The vacuum in the He Transferline may break and with it the vacuum in the Coldbox since these to chambers form one volume.
j.3	Remove the **protective caps** from the CryoCouplers on He Transferline and CryoProbe.

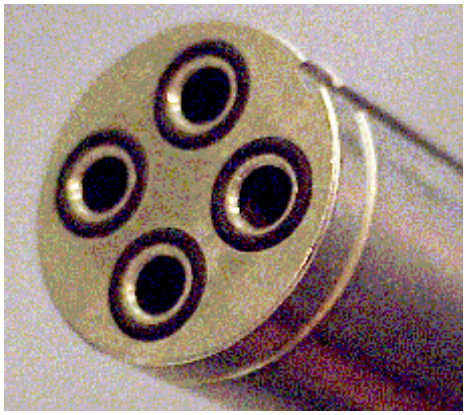
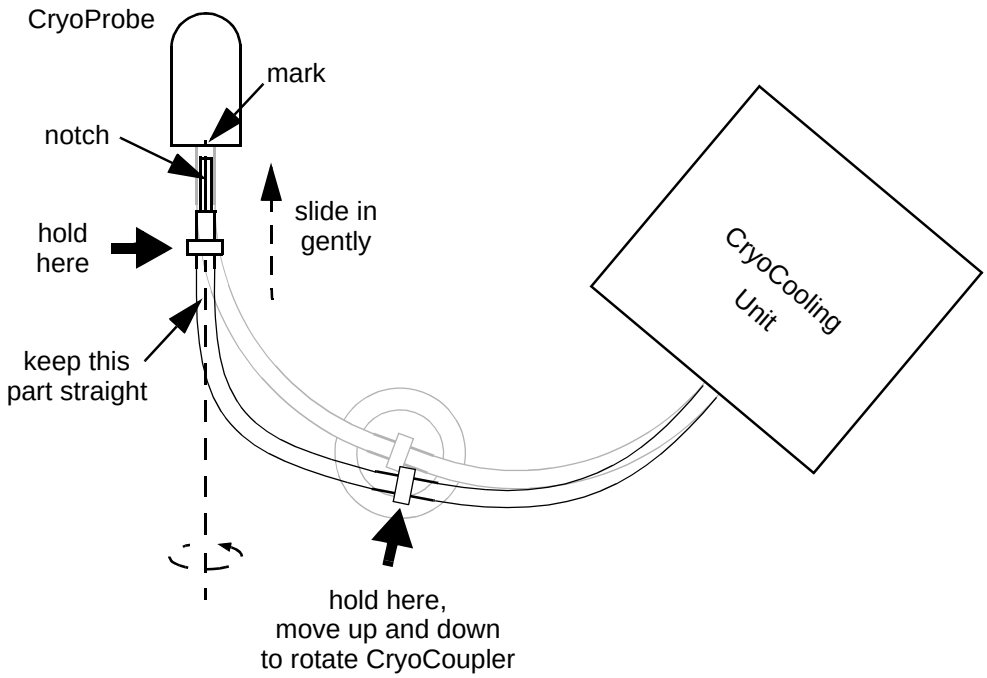
j.4	Check the **four o-rings** on the He Transferline CryoCoupler (***Figure 3.13.***): are they in place, clean and undamaged? If not replace with the o-rings found in the spare parts box delivered with each CryoProbe System. The o-rings are of type viton and size 7.1 by 1.6 mm. *Figure 3.13. O-rings on the CryoCoupler* 
j.5	Turn the CryoCoupler end such that it will fit into the CryoProbe. The **groove** must be exactly on **top**. Do not bend the Transferline while twisting. **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.
j.6	Hold the **CryoCoupler** on the He Transferline with **one hand**. Take the **vacuum joint** which is 1 m away from the CryoCoupler into the **other hand** (see ***Figure 3.14.***).

Figure 3.14. 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	**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! Slide the CryoCoupler **into the CryoProbe**. It should fit smoothly without any significant movement of the suspended magnet. **CAUTION:** If the CryoCoupler gets stuck, do not force it into position. Do not bend the He Transferline to an extreme shape to make it fit - permanent damage on CryoProbe or He Transferline could result! The He Transferline Support may be in the way during the insertion process. If this is the case, remove the CryoCoupler, move the Transferline Support a little aside and repeat the insertion process. When a smooth insertion fails, take the CryoCoupler out and try again.

j.9	Use the special open-end **titanium wrench** to fix the CryoCoupler gently to the CryoProbe. Take care not to damage any connectors on the CryoProbe front. Hold the He Transferline end with your other hand to take up the torque applied.
j.10	Adjust the height of the **Transferline Support** in such a way that it has the same level as the connector of the CryoProbe. Position the Transferline Support such that the He Transferline falls exactly (to within 1 mm) into its bed. If there are more than one Transferline Supports, adjust the height and position of the others in order to achieve a smooth bending curve of the Transferline.
j.11	Make sure that the **CryoProbe is still centred** inside the (optional) cranked pillar braces. If not, relocate the Transferline Support accordingly. **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.
j.12	Tighten all **screws on the vacuum flange** very carefully in a balanced fashion to align the seal inside properly. **NOTE:** If a subsequent cool-down fails due to vacuum problems, the vacuum flange may not have been closed properly.
j.13	**Lock the He Transferline** on the Transferline Support with the four black screws.

Load firmware *3.15*

The laptop has the necessary software and directory structure already 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:

`\\Bruker\unitool`	*UniTool* program
`\\Bruker\unitool\files\crco`	*Firmware* file `crcoxx.abs`
`\\Bruker\unitool\files\temp`	
`\\Bruker\CryoTool`	*CryoTool* program
`\\Bruker\CryoTool\LogFiles`	logfiles

REMARK: The superior directory is either `D:\app or C:\app or C:\.`

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 `\\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 `\\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 `\\Bruker\unitool`. The *LabWindows/CVI Run-Time Engine Directory* should be the default `\\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 `\\Bruker\unitool\files\crco` to the CryoController. It lasts for a couple of minutes and ends with an automatic reboot of the CryoController.

Short-cuts

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 `\\WINNT\Profiles\All Users\Desktop\Bruker` - right-click `CryoTool` - **Properties** - **Shortcut** - **Start in:** `\\Bruker\CryoTool\LogFiles`. Repeat this procedure for ...`\Start Menu\`... instead of ...`\Desktop\`... .

Hook up the He steel-cylinder *3.16*

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

Necessary tools:

1open-end wrench $1^{5}/_{16}$”

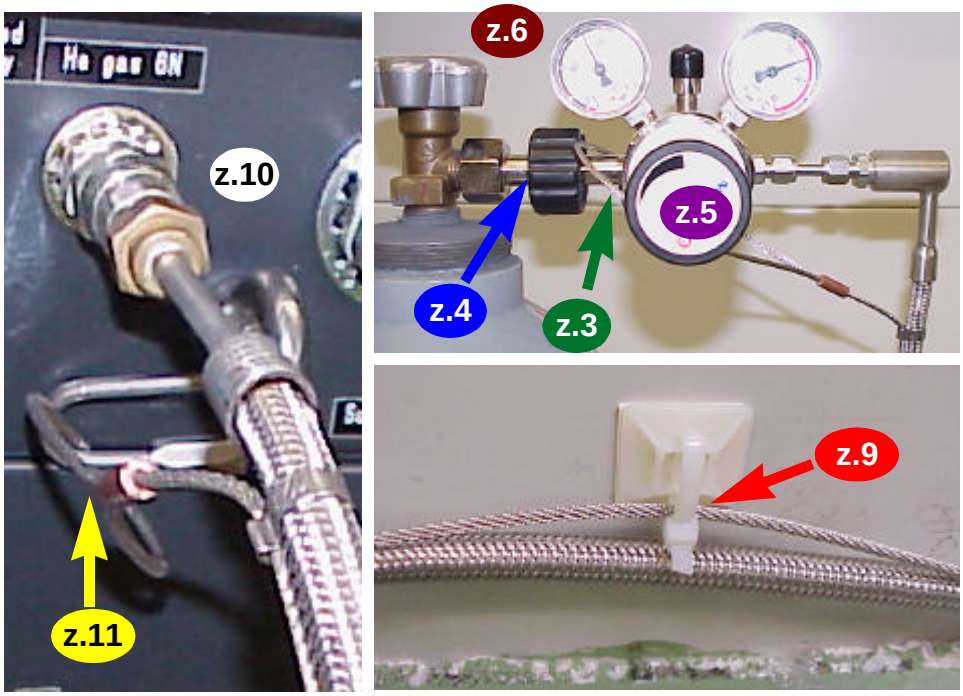
1open-end wrench $^{5}/_{8}$”

1open-end wrench $^{3}/_{4}$”.

Table 3.10. Mount the He steel-cylinder

step	action
z.1	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. **WARNING:** The He steel-cylinder and its entire transport path must always be outside the 0.5 mT range of the magnet.
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 3.15.***).

Figure 3.15. 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 using the wrench $1^{5}/_{16}$”.
z.5	Open the He Regulator by about two turns and connect the dump tool to the other end of the He Hose.

z.6	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.7	Wait about 10 seconds then 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.8	1st leak test: **mark** the actual pressure on the primary gauge and **close** the He steel-cylinder main valve.
z.9	**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. Attach the He Hose steel wire at **intervals of about 1 m** firmly to the wall or to the floor. **WARNING:** Avoid the use of magnetic materials or tools inside the 0.5 mT stray field of the magnet.
z.10	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.
z.11	Lock the **steel cable loop** in the hook that is provided at the CryoCooling Unit back-panel.
z.12	End of 1st leak test: (Must be done at least half an hour after step ***z.8***.) 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. Take snoop liquid to locate the leak.
z.13	Open the He steel-cylinder **main valve** by ~one turn.
z.14	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. **IMPORTANT:** Never exceed 30 bar - the gauge would be damaged! 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.14***.
z.15	2nd leak test: Wait at least 10 minutes. 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. **NOTE:** The 2nd leak test will be terminated in ***"Interconnect the He Compressor" on page 40***, step ***i.13*** below.

Interconnect the He Compressor *3.17*

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:

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

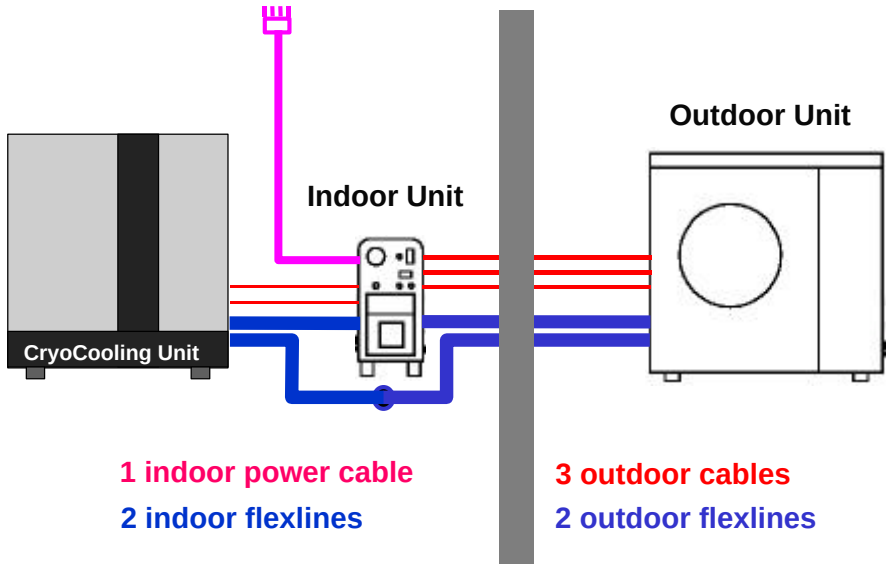
Table 3.11. Interconnect the He Compressor

step	action
i.1	**IMPORTANT:** The o-rings and rubber gaskets on all Flexline connectors must be very clean and properly positioned. 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. The rubber gaskets should have a small grease layer on the surface. If it has been removed put some vacuum grease on them.
i.2	**CAUTION:** Connect the Flexlines first at the **He Compressor**. **Loosely attach** the Flexlines to the GAS port on He Compressor. Each Flexline is symmetric, i.e. there is no preferred orientation. **CAUTION: Do not interchange** SUPPLY **and** RETURN. The Coldhead would become inoperable when switched on and can only be repaired through complicated service procedures!
i.3	When **locking** the Flexlines, make the first turns by hand. Then, use open-end wrenches 1" and 1 3/16" on the Flexline's two terminal hexagon nuts. After reaching the stop, rotate back by a ¼ turn to facilitate eventual unlocking. **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.
i.4	Fix the other end of the Flexlines to the **CryoCooling Unit**'s ports. If you install an **outdoor He Compressor**, connect the indoor and outdoor flexlines according to ***Figure 3.16.***

i.5	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. **IMPORTANT:** If the pressure gauge indicates plain 0 bar, contact BRUKER: this is a major problem and a lengthy gas purifying cycle is required.
i.6	If the He Compressor requires water-cooling, connect it to the in-house **cooling water** cycle or to the optional water chiller. **NOTE:** The installation of the water cooling system is not subject of this manual. Turn on the water and check for leaks.
i.7	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.
i.8	Plug the 4-pin **Coldhead power cable** 45446 (20m) / Z14113 (6m) into COLD HEAD POWER on the CryoCooling Unit and COLDHEAD JC on the He Compressor. **CAUTION:** Never connect the Coldhead cable between He Compressor and Coldhead while the He Compressor is energized.
i.9	Connect the **He Compressor control cable** Z13883 (20m) / Z14323 (6m) from the **CryoCooling Unit** to EXTERNAL JR on the **He Compressor**. Set the *REMOTE DRIVE* at the He Compressor to *EXT* position. **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.
i.10	Note down the values from the **hour meters** on the He Compressor front and the CryoCooling Unit back into the Service Logbook (Z70590).
i.11	**Outdoor He Compressor only:** Establish the electrical connections between the outdoor and indoor unit of the He Compressor according to ***Figure 3.16.***. Connect each individual wire of the power cable and the two control cables to the corresponding socket. **CAUTION:** Make sure that the mains power cable is not jet connected.
i.12	**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. Plug-in the He Compressor but do **not** switch it *ON* yet. **Outdoor He Compressor only:** Make a function test according to ***Table 3.3. Function test***

i.13	End of 2nd leak test: 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.
i.14	Open the He steel-cylinder **main valve** by ~one turn.

Figure 3.16. Connections Outdoor He Compressor



Warning labels *3.18*

Stick a warning label (***Figure 3.17.***) on all mobile units of a CryoProbe System that contain magnetic materials and could be attracted by the NMR magnet.

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 3.17. 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!

Figure 3.18. Overview of the connections (without HPPR and RF-cables)

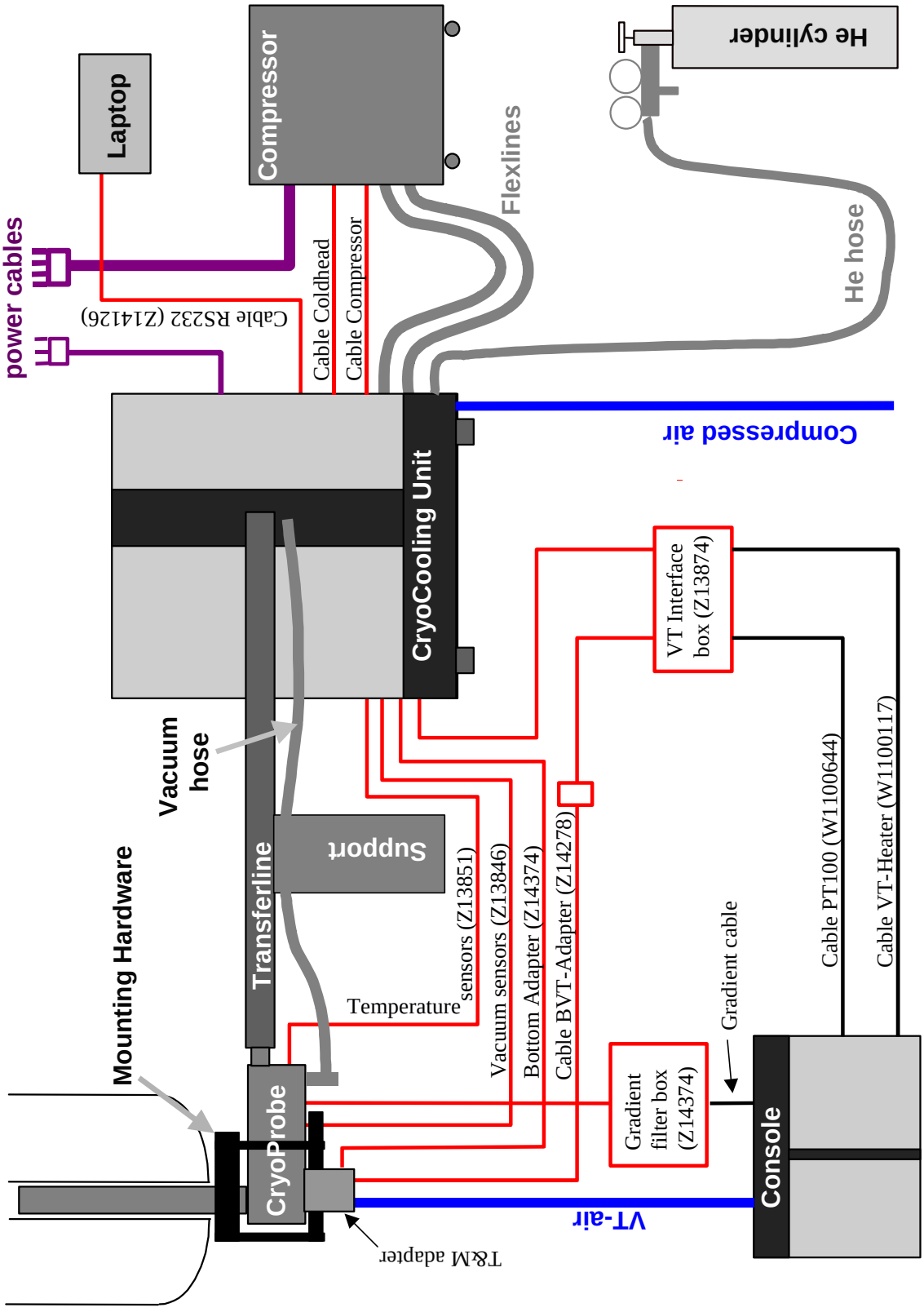


Figure 3.19. CryoProbe connections

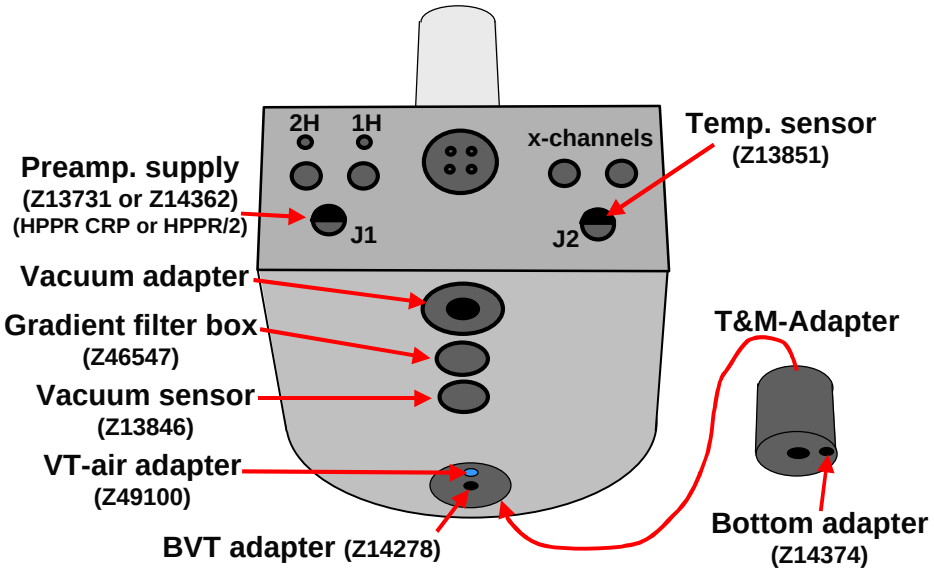


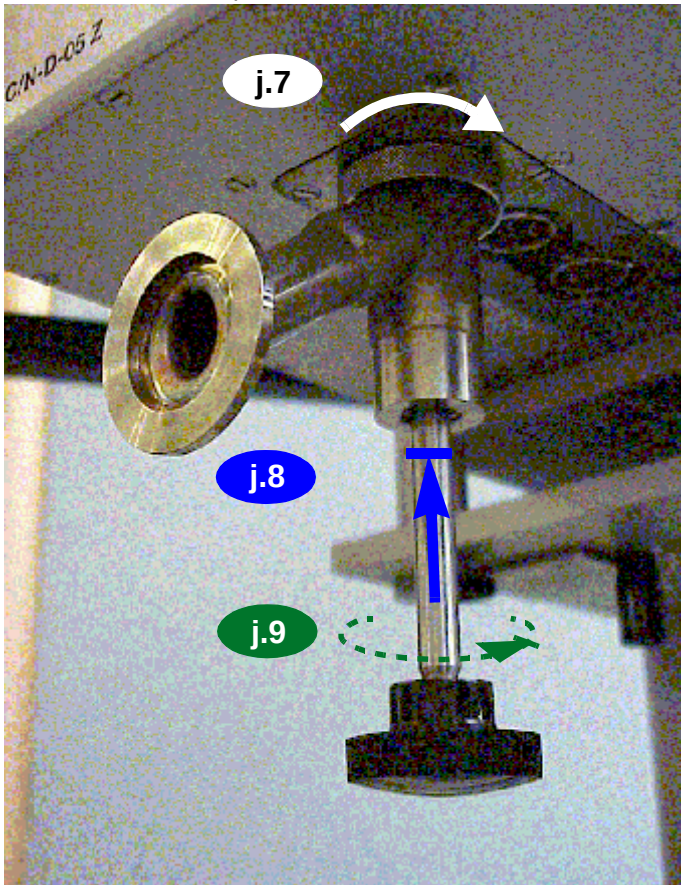
Table 3.12. Connect the CryoProbe

step	action
j.1	Plug the vacuum and temperature **sensor cables** from the CryoCooling Unit into the CryoProbe bottom and into J2 on the front, respectively. Connect the CryoPreamp supply cable from the HPPR CRP or HPPR/2 to J1.
j.2	Connect the **VT sensor cable** and the **VT gas hose** to the probe bottom. Connect the heater cable (W1100117) and the PT-100 sensor cable (W1100644) of the BVT to the VT Interface Box. At the VT unit, e.g. B-VT3000, the Pt100 sensor must be selected. The reading in ***edte*** should indicate **room temperature**.
j.3	Link the BVT cable from the **VT Interface Box** to the BVT socket on the **CryoController** inside the CryoCooling Unit.
j.4	Extent the other line from the **VT Interface Box** with cable Z14278 which will go to the **CryoProbe**.
j.5	Attach the **Tuning Adapter** 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.
j.6	Connect the **Bottom Adapter** cable to the Tuning Adapter.
j.7	Mount the **Vacuum Adapter** to the CryoProbe bottom such that it points to the front. Connect the **vacuum tube**.

Table 3.12. Connect the CryoProbe

step	action
j.8	**Push in** the actuator screw of the Vacuum Adapter (black handle) in as far as it will go (***Figure 3.20.***).
j.9	**Turn** the actuator screw **clockwise**. Its thread must grip into the thread of the CryoProbe's vacuum plug. Make sure the handle is slightly moving up while screwing. Turn it about one and a half turn inside. Try not to turn it until the stop is reached. **ATTENTION: Do not jet draw out** the CryoProbe's actuator screw. Do this later, during the cool-down procedure when being required by the software.

Figure 3.20. Vacuum Adapter



j.10	Connect the **remaining RF and gradient cables** (for details, see *CryoProbe RF Electronics Technical Manual*).
j.11	Regulate the **VT gas flow rate** to (at least) the value specified on the LIMITATIONS - WARNINGS sheet.
j.12	Set the **VT heater power limit** as given on the LIMITATIONS - WARNINGS sheet.

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"*** 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 N_2 gas and dry air can be used as VT gases.

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	**Connect the VT gas** inlet of the CryoProbe either directly to the VT unit or to a BCU05 gas cooler but do not switch the BCU05 *ON* yet. **CAUTION:** When cold operation has been started, **the VT gas supply must not be interrupted** before the CryoProbe has been warmed-up again. Thus, a BCU05 gas cooler can only be inserted while the CryoProbe is warm.
v.2	**Set** the VT gas **flow rate** and VT **heater power** limit according to the values given on the specific LIMITATIONS - WARNINGS sheet of the CryoProbe. Do **not** switch the VT heater ***on*** yet. **NOTE:** A high VT gas flow rate is recommended but it must not lift the sample.
v.3	Verify that there is **no sample in the magnet yet.**

v.4	**Configure** the VT unit for the **Pt100** temperature sensor with ***edte***. Make sure the Pt100 sensor cable is connected properly at CryoProbe and VT unit. **NOTE:** An optional **B-TO2000** unit for conventional probes uses the same type of Pt100 cable. The cable is suitable for the CryoProbe and there are no special considerations when using a B-VT3000. A **B-VT2000**, however, cannot be toggled properly from B-TO2000 to CryoProbe operation with ***edte*** (although no error occurs); it must be **set explicitly at its EUROTHERM** module. With a B-TO2000, the EUROTHERM would be set neither for a Pt100 (code 0225) nor for a Cu/Const thermocouple (code 0203) but with the special code 5203. When using a CryoProbe, make sure the EUROTHERM is configured for Pt100 with **code 0225**. See the *B-VT2000 Operating Manual* (P/N W1101034) for instructions.
v.5	Set the **sample temperature** to somewhere above room temperature, e.g. ***300 K***.
v.6	Switch the VT heater ***on***.
v.7	**Wait** until the 'sample' temperature and the heater power in the ***edte*** window settle.

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

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

Basic tests before cool-down *3.21*

Before the first cool-down is started during the installation of a CryoProbe System, it is recommended to go through a few fundamental tests. The firmware of the CryoController is surveying all sensors of the system and will abort the cool-down procedure and display error messages when abnormalities like leakages are detected. (See the *Service Manual* for details about error messages.) Repeated automatic abortion of the cool-down can be avoided when going through the following tests saving time and He gas.

Power-on *3.21.1*

If the CryoProbe System is freshly being installed or has been *OFF* for a longer period, 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 back-panels of CryoCooling Unit and He Compressor.

Table 3.14. 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	Check that the **He Compressor** *MAIN POWER SW* at its backside is switched ***ON*** and that the ***DRIVE*** at its front is *ON.* The unit is in the **stand-by** mode. It is started later by the CryoController. On the backside, *COLDHEAD DRIVE* must be *OFF* and *REMOTE DRIVE* in *EXT* position (see ***"Figure 3.9."***). **CAUTION:** If *COLDHEAD DRIVE* and *REMOTE DRIVE* are in the wrong positions, the Coldhead inside the CryoCooling Unit can be damaged.
o.6	Reboot the CryoCrontroll by turning *OFF* and again *ON* the **CryoCooling Unit** main switch at its front. The CryoController will initialize the CryoPlatform. After a few moments the **green *WARM*** and the **white *UNPLUG* must be on**. If *ERROR* lights up, try to reset it by pushing *WARM UP* and check if all sensor and supply connections are ok. **REMARK:** When the CryoController is re-booted, the BVT-heater must be switched *ON* again!
o.7	Re-initialize ***CryoTool*** on the laptop by closing and re-starting it. No error messages should appear. **NOTE:** If the CryoTool is started before the CryoCooling Unit is switched *ON*, it will go to off-line mode because it sees no response at its *COM1* port.

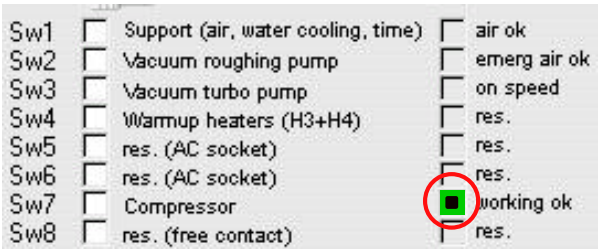
Test the He Compressor and Coldhead *3.21.1*

In the CryoTool Service window check that the indicator ‘working ok’ is on.

If not check the pressure inside the He Compressor. If its less than 1.5bar, then do the function test only after ***"Refill the He Compressor (if necessary)" on page 50***.

If the indicator ‘working ok’ is still not on, check the He Compressor control cable as described in the *Service Manual*.

Figure 3.21. ‘working ok’



Activate Sw7 Compressor for a short moment. The He Compressor should start and the hissing sound of the Coldhead should become audible. Do not forget to switch off Sw7 after the test.

Test the vacuum *3.21.2*

Starting conditions

1. Since the vacuum valves are working pneumatically make sure that compressed air pressure is within the required pressure limits. The indicator ‘air ok’ should be on.
2. The turbo pump is on speed and the coldbox and CryoProbe have been evacuated for a long time and the readings of the Sensors VacCRP and VacCU are at least 5.5 mV and stable as described in ***"Start to evacuate the Coldbox" on page 20***.

Procedure

1. Close both valves V11 and V12.
2. The vacuum readings should remain stable.

If one of them is dropping, then either there is a leakage or there is still moisture inside the system. In the second case, it needs more time to evacuate the Coldbox and CryoProbe. If there is a leakage, check the O-rings in the vacuum piping and especially the vacuum operator. A typical leakage of the Coldbox vacuum is the vacuum flange in the Transferline which has been opened during the adjustment of the CryoCoupler (see ***"Joining the CryoCoupler" on page 33***).

For further troubleshooting see the corresponding section in the *Service Manual*.

Test the Helium piping *3.21.3*

Starting conditions

The Helium hose has passed the previous leakage test and all valves in the Cryo Service window are on the left position.

Table 3.15. Test the Helium piping

step	action
i.1	Make sure the He cylinder main valve is opened and that the secondary pressure is about 22 bar.
i.2	Close V4 and V8 (right position, since they are normally opened)
i.3	Open V2, V3 and V5. Wait a few seconds until the reading of HeSupp and HeRet are about 22bar.
i.4	Close V5.
i.5	Observe the reading of HeSupp and HeRet. After a while they should remain stable and no dropping should be observable. Otherwise a Helium leakage must be located and fixed (see the *Service Manual* for detailed troubleshooting).
i.6	If the test is passed, reboot the CryoController to reset the system in its default position.

Refill the He Compressor (if necessary) *3.21.4*

If the pressure inside the He Compressor is < 10bar, then the automatic refill will-not be carried out. Therefore a manual refill will have to be done.

Table 3.16. 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. Following conditions must be fulfilled: 1) cooling water supply is active (if applicable); 2) He steel-cylinder is connected and open. Its primary pressure must be larger than 40 bar and its secondary pressure stable at 22-25 bar; 3) CryoProbe is properly installed and connected; 4) CryoCooling Unit is *ON* and *WARM* and *unplug* indicate that the unit is ready; 5) He Compressor *MAIN POWER SW* at its backside and *DRIVE* at its front are *ON*.
f.2	Start a *COOL DOWN* and wait until it stops with the error 235 ‘Compressor pressure much too low. Probably leaking.’.
f.3	Reboot the CryoCooling Unit. Wait until all valves are on the **left**.
f.4	Enter the password-protected ***Service*** mode of the ***CryoTool***.
f.5	In the ***CryoTool***, close ***V1, V2, V3, V4*** and ***V8***.

f.6	Open ***V6***. **WARNING:** Never open ***V6*** or ***V7*** without having flushed the system first after having opened the CryoCoupler!
f.7	Now the timing is important: Open ***V5*** for a few seconds and close it again. Wait until the ***HE-Supp.*** reading gets stable. If ***HE-Supp.*** is still lower than 17.5bar, open again ***V5*** for a moment. Do this until 17.5bar are reached. If the reading exceeds 17.5bar, dump the excessive He by opening ***V7***.
f.8	Close all valves. The system is ready for *COOL DOWN*.
f.9	Exit the service mode.

Cool-down *3.22*

NOTE: If any detectable malfunction appears, a cool-down will be automatically stopped and the CryoProbe System is automatically warmed-up to ambient temperature. See the corresponding section in the *Service Manual* for the meaning of error messages and troubleshooting.

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.

Go through the cool-down check list before starting the cool-down.f

Table 3.17. Cool-down

step	action
c.1	Set the cool-down timeout to a higher value than the default value. (See also the corresponding chapter in the *Service Manual*. 1.) Close CryoTool 2.) Start UniTool and select the option 6 Get/Set Cooldown timeout in the Configuration Menu. 3.) change the default value from 270min to i.e. 1000min. and exit UniTool. This will give you more time to do flow adjustments (***"Adjust the flow (if necessary)" on page 55***).
c.2	**Start** the cool-down by pushing the *COOL DOWN* button on the Cryo-Cooling Unit or in the ***CryoTool***. *COOL DOWN* will start to flash, indicating that the system is preparing the cool-down process. If the He Compressor had to be refilled manually as described in ***"Refill the He Compressor (if necessary)" on page 50***, the option *NO FLUSH COOL-DOWN* can be selected in the Service mode in order to save time and Helium. The option *NO FLUSH COOL-DOWN* may not be selected, if the CryoCoupler has been disconnected since the last flushing cycles. A No Flush Cool-down will further more only be accepted, if the vacuum of CRP and CU are above 5.5mV and if the turbo pump is on speed.

c.3	*COOL DOWN* will keep flashing as long as the ambient temperature preparations (evacuation, flushing cycles and charging with He) are in progress. Several pneumatic **pops** will be audible. At this point, it is recommended to check the secondary pressure at the He Regulator: it should always come back to **22-25 bar**. The primary pressure must remain ≥ 40 bar at all times.
c.4	Open the manual Vacuum plug as soon as the software requires it in the CryoTool main window.
c.5	~15 min later, **CryoCooler and He Compressor** will be **started automatically** to perform the cool-down. *COOL DOWN* changes to steady white during the actual cool-down. The characteristic **periodic hiss** of the CryoCooler will be audible then.

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

When cool-down has started, ***"Load additional Xwin-nmr software (up to 2.5)" on page 53*** and ***"Put HPPR CRP or HPPR/2 assembly into operation" on page 53***. The HPPR CRP or HPPR/2 can further more be configured according to the *User Manual*.

c.6	**If *COLD*** on the CryoCooling Unit **flashes**, it will take about half an hour more until the final temperature is reached and stabilized. **NOTE:** A cool-down must not take more than 4 h; if it does, the flow will need some adjustment (***"Adjust the flow (if necessary)" on page 55***).
c.7	**Verify** that the **'sample' temperature** reading from inside the cavity is at its preset value and stable. Now a **sample can be inserted**. At this point, the ***"Low temperature limit in the sample cavity"*** as described in the *User Manual* should be determined. **NOTE:** For recommended sample depth and filling height, see the 'LIMITATIONS - WARNINGS' sheet for the CryoProbe and ***"Samples and spinners"*** in the *User Manual*. **IMPORTANT:** Always keep an eye on the sample temperature with ***edte*** when working with the CryoProbe. If the temperature drops, eject the sample and keep monitoring the 'sample' temperature inside the CryoProbe cavity.
c.8	**Shimming** can be started while *COLD* is still flashing, see ***"Shimming and Lock-in"*** in the *User Manual*.
c.9	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 (up to 2.5) 3.23

For Xwin-nmr up to 2.5 and HPPR CRP the AU-programs ***crpon***, ***crpoff*** etc. have to be installed 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.

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 or HPPR/2 assembly into operation 3.24

NOTE: The HPPR CRP or HPPR/2 can be installed conveniently while the CryoProbe undergoes its first cool-down.

HPPR CRP 3.24.1

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*)

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 3.18. 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 *CryoProbe RF Electronics Technical Manual* 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 *JU*2 is encountered in the non-default position on the existing HPPR, it is likely that the corresponding jumper *JU20* 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 *17* and *18*); whereas at the **15N CRP module** the lower two pins should be connected (*18* and *19*).
e.6	**Switch *ON* the AQR** and run an XWIN-NMR ***cf*** dialogue. The first X-module in the HPPR CRP will be recognized as ***X-BB31P_2HS*** and an optional second module as ***X-BB19F_2HS***. If more than 2 modules for X-nuclei are active, the ***cf*** dialogue may run into an error.
e.7	Run ***xau crpwobb*** in XWIN-NMR 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.

HPPR/2 ***3.24.2***

NOTE: See also in the B.A.S.H. CD from version 4 on or in the HPRR/2 Technical Manual (Z31559).

The HPPR/2 is fully CryoProbe compatible. It needs a software off at least XWin-NMR 3.0. From XWin-NMR 3.5 on the software will read Probe datas from PICS and automatically set the configuration for a CryoProbe or for an ordinary probe.

For older software version the configuration needs to be set manually as the following:

Open a Unix shell and type:

`xwinnmr -e UniTool hppr` .. ***`2x return`***
`p` (Preamplifier modules) ..... ***`... return`***
e.g. **`5`** (for 1H) ..... ***`... return`***
`s` (set force state) ..... ***`... return`***
`4` (=cold) ..... ***`... return`***
`x`..... ***`... return`***
`x`..... ***`... 2x return`*** to exit

Then type **`ii`** in the XWin-NMR window to initialize.

The other channels can be set accordingly.

When wobbling, the force state does not need to be set off.

NOTE: After a power up of the HPPR/2 the force states have to be set again.

Adjust the flow (if necessary) *3.24.3*

At most systems the flow must undergo some fine tuning. See the section "***Gas-flow***" in the *Service Manual*.

How to proceed *3.25*

When the CryoProbe is cold and ready, continue according to the *CryoProbe System User Manual*.

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 ***pl*** values depends on the actual spectrometer hardware, the RF limits must be translated into power levels ***pl***. If CORTAB is used on the spectrometer, determine them with the actual CORTAB and - for safety - without CORTAB. Write these ***pl*** 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 ***pl=-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)

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 that 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*** < ~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 H_2O 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 65***.

Relocation of a CryoPlatform

5

Relocation 5.1

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 60***.

For disassembling the CryoProbe System, use the reverse order of the installation sequence ***"Initial setup" on page 13***. 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 5.2

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 5.3

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

Pressure release *5.3.1*

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 5.1.*** and let the gas escape. Leave the nuts unlocked but keep the connectors on their threads.

Figure 5.1. Releasing the gas from the CryoCooling Unit

Toxic materials *5.3.2*

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.

Relocation of a CryoPlatform

Checklists

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?
- ❑ Optional: Is the acoustic insulation for the He Compressor and the Flexlines 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.
- ❑ Are the HPPR CRP or the HPPR/2 or some of its modules at the site? Will the boards and cables needed for a possible upgrade of the console be at hand?

Quick installation guide *1.3*

- ❑ Unpack and check for completeness.
- ❑ Position the CryoCooling Unit and start the evacuate the Coldbox.
- ❑ Test the He Compressor.
- ❑ Position all other units according to the site planning.
- ❑ Modify the magnet if necessary (shim system, pillar braces).
- ❑ Fix the Mounting Hardware.
- ❑ Insert the CryoProbe into the magnet.
- ❑ Make a sample spinning test.
- ❑ Join the CryoCoupler and connect the Transferline.
- ❑ Connect the He Cylinder, purge it and start the leakage test.
- ❑ Connect the Flexlines.
- ❑ Connect all cables.
- ❑ Setup the VT-air and -heater.
- ❑ Do the basic tests before Cool-down (see foll ***"Check list before Cool-down" on page 65***).
- ❑ Fill the He Compressor after the automatic flushing if necessary.
- ❑ Download latest firmware.
- ❑ Start cool-down.
- ❑ If needed, download the necessary Xwin-nmr software and perform the HPPR CRP or HPPR/2 setup.
- ❑ Adjust the He flow after the thermal equilibrium of all temperatures are reached.

Check list before Cool-down *1.4*

The following topics should be checked before Cool-down can be started:

- ❑ **cooling water** supply is active (if applicable).
- ❑ He steel-cylinder is connected and opened. Its primary pressure must be larger than 40 bar and its secondary pressure stable at **22-25 bar**. The He adapter and hose have passed the leakage test.
- ❑ *SUPPLY PRESSURE* gauge on the **He Compressor** front reads about **17.5 bar** (= 1.75 MPa or 17.9 kgf/cm^2). If the pressure is higher, the excess He will be released automatically during the cool-down preparations.
- ❑ **He Compressor** *MAIN POWER SW* at its backside and *DRIVE* at its front are ***ON***.
- ❑ **He Compressor** has passed its function test (Phasing and voltage correct).
- ❑ CryoProbe is properly installed and connected.
- ❑ CryoCooling Unit is *ON* and ***WARM*** **and *unplug*** indicate that the unit is ready.
- ❑. Indicator ***air ok*** (Service Window of CryoTool) is on.
- ❑ Indicator Compressor ***working ok*** (Service Window of CryoTool) is on.
- ❑ Pumps are working properly (Indicator turbo pump ***on speed*** (Service Window of CryoTool)) and there is no vacuum leak.
- ❑ He piping has passed the leak test.
- ❑ 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.
- ❑ Verify that there is **no sample in the magnet yet.**

User training *1.5*

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 or HPPR/2 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, intervals between two required cool-down/warm-up cycles, etc.
- ❑ User maintenance (He steel-cylinder exchange, etc.) and periodic service by BRUKER.
- ❑ BRUKER support and service: addresses, error messages, logfile, fault report, repair declaration, description of circumstances etc.
- ❑ Shutdown and storage.

Related documents A

The following documents contain further information.

CryoProbe System Ordering Information

A list of components and options with hints on how to place a complete and consistent CryoProbe System order.

CryoProbe Site Planning Questionnaire

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

CryoProbe System Site Planning Guide (P/N Z31524)

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

CryoProbe System Site Preparation Manual (P/N Z31553)

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

CryoProbe System User Manual (P/N Z31551)

Contains all important information for the user and is also useful when customizing the CryoProbe system during the installation

CryoProbe System Service Manual (P/N Z31677)

This manual contains important information when troubleshooting the CryoProbe system and when performing an maintenance visit. It should also be at hand when doing an installation.

CryoProbe data sheets

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

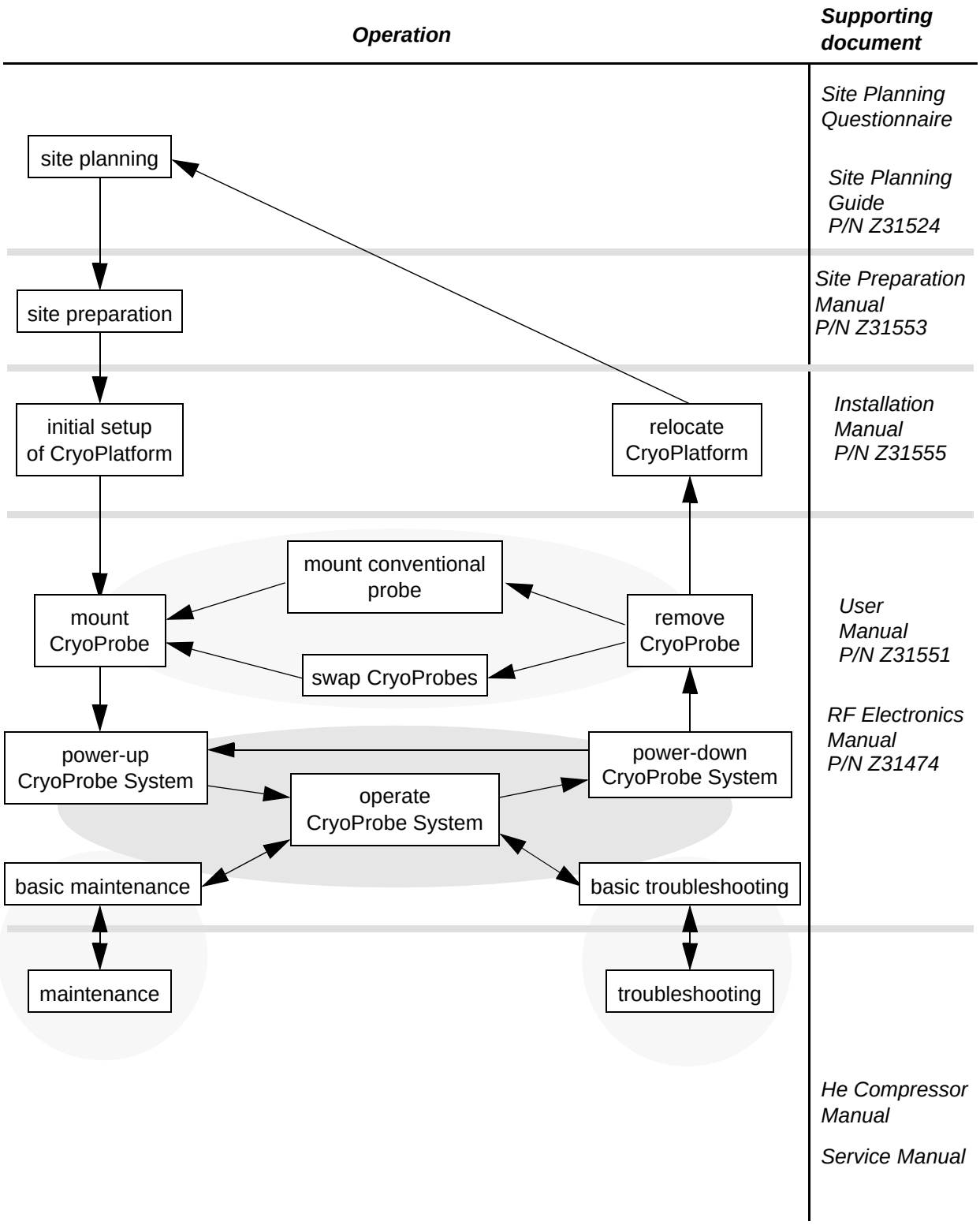
He Compressor technical manual

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.

Figure A.1. When to use which CryoProbe document



Conversion of metric units

B

1 bar ≡ 0.1 MPa

1 bar ≈ 14.5 psi

1 bar ≈ 1.02 kgf/cm^2

1 Pa ≡ 0.01 mbar

1 psi ≡ 68.95 mbar

1 kgf/cm^2 ≈ 0.98 bar

1 kg ≈ 2.2 lb

1 lb ≡ 0.4536 kg

1 mm ≈ 0.04 inch

1 m ≈ 3.28 feet

1 inch ≡ 25.4 mm

1 foot ≡ 0.3048 m

1 Nm ≈ 8.85 lbf-inch

1 lbf-inch ≈ 0.113 Nm

1 L (liter) ≈ 0.264 gallon (U.S.)

1 L (liter) ≈ 0.220 gallon (Brit.)

1 gallon (U.S.) ≈ 3.79 L

1 gallon (Brit.) ≈ 4.55 L

1 kWh ≈ 3.6 MJ

1 kWh ≈ 3412 btu

1 MJ ≈ 0.278 kWh

1 btu ≈ 0.293 Wh

1 mT ≡ 10 Gauss

1 Gauss ≡ 0.1 mT

°C to °F:

$T_{°F} = (T_{°C} \times 1.8) + 32$

°F to °C:

$T_{°C} = (T_{°F} - 32) / 1.8$

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

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

Conversion of metric units

Glossary

C

Adsorber

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

BBIS

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

Coldhead

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

CryoController

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

CryoCooler

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

CryoCooling Unit

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

CryoCoupler

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

CryoPlatform

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

CryoPreamp

A cryogenically cooled preamplifier module inside the CryoProbe housing. There is a frequency-specific preamp module for each channel of a CryoProbe. A CryoPreamp cannot be separated from its CryoProbe. It always requires an additional external HPPR assembly, the cryo-compatible HPPR CRP or an HPPR/2.

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. A HPPR/2 is equipped for the CryoProbe by default. 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 **I**dentification and **C**ontrol 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 **q**uality factor Q is a measure of the efficiency of reactive devices such as inductors, capacitors, or resonant circuits.

Radiator

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

RF

Radio **f**requency

Transferline Support

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

Tuning Adapter

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

Tuning Tool

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

UniTool

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

UPS

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

Vacuum Adapter

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

Vacuum Plug

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

Vacuum bellows

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

Vacuum system

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

VT gas

Usually nitrogen gas or dry air at a controlled **v**ariable **t**emperature that flows through a probe to heat or cool the sample. Its function must not be confused with

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

VT Interface Box

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

VT unit

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

Water chiller

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

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

Glossary

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