



# **SPECTROSPIN NMR Magnet System**

## **SPECTROSPIN NMR Magnet System Trouble Shooting**

**Version 002**

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**BRUKER**

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## 7 Trouble Shooting

### 7.1 Trouble shooting during Assembly

<b>Problem indicators</b>	<b>Possible reasons</b>	<b>Solutions</b>
Tilt indicators and/or shock watches are broken.	Uncareful transportation or transportation accident.	Contact shipping agent, check the whole magnet system for damages.
Visible damages.	Uncareful transportation.	Contact shipping agent, check all tilt indicators and shock watches.
Ceiling height too low for assembly on magnet stand.		Do the assembly on the assembling feet.
Ceiling height too low for exchange of helium level sensor.		Insert helium level sensor while magnet system is on the assembling feet.
He vessel and N <sub>2</sub> vessel are not concentric.	Alignment is not done correctly.	Repeat the alignment of the He vessel and the N <sub>2</sub> vessel.
	Alignment rod is lose.	Check fixation of the alignment rods.
	Alignment rod is broken.	Replace defective alignment rod.
	Reduction flanges may be non concentric.	Check orientation of the non concentric reduction flange.
N <sub>2</sub> vessel and OVC are not concentric.	Reduction flanges may be non concentric.	Repeat alignment of the N <sub>2</sub> vessel and the OVC.
Vacuum in OVC does not reach 10 <sup>-5</sup> mbar within 48 hours.	O rings may be leaky	Check vacuum tubing to the vacuum pump. Check O rings in the vacuum valve. Check O rings and sealing surfaces on reduction flanges and on sealing flanges. Check O ring and sealing surfaces of the drop off plate.

<b>Problem indicators</b>	<b>Possible reasons</b>	<b>Solutions</b>
Vacuum in OVC does not reach $10^{-5}$ mbar within 48 hours.	Room temperature bore tube has scratches or dust on the sealing surfaces.	Check sealing surfaces on the room temperature bore tube: No scratches and no dust must be visible!
	Moisture may have entered the OVC during transportation or during assembly.	Pump and flush the OVC several times with dry nitrogen gas to remove moisture.
	Pump stand is defective and does not reach the required vacuum of $10^{-6}$ mbar.	Check pump stand. Pumping directly against a sealing cap produces pressure below $10^{-6}$ mbar.
Upper pillar brace collides with vacuum valve.	Vacuum valve is not orientates parallel to the pillar brace.	Turn the vacuum valve until the flange does not collide with the pillar brace any more. This operation may be carefully done with evacuated OVC.
Superisolation touches OVC or radiation shield or bore tubes.	Superisolation was not fixed correctly during assembly.	Fix the superisolation on the N <sub>2</sub> vessel properly with the supplied polyester tape. Avoid any connection between different vessels or bore tubes in the cryostat carefully!

## 7.2 Trouble shooting during Cool Down Procedure

Problem indicators	Possible reasons	Solutions
Precooling with liquid nitrogen proceeds too slowly.	Transport dewar for liquid nitrogen is empty.	Refill transport dewar.
	Transfer pressure is too low.	Increase transfer pressure.
	Transport dewar is leaky, no transfer pressure may be applied.	Check transport dewar for leakage and seal gas supply tubing.
Precooling with liquid nitrogen proceeds too fast.	Transfer pressure is too high.	Immediately stop precooling and adjust transfer pressure properly.
	Large coils must be pre-cooled with ACD* (Automatic Cooling Device).	Connect ACD* correctly to the NMR magnet system for cool down procedure.
Vacuum in OVC does not reach $10^{-6}$ mbar during pre-cooling.	O rings may be leaky.	Stop precooling and warm up the NMR magnet system. Check vacuum tubing to vacuum pump. Check O rings in the vacuum valve. Check O rings and sealing surfaces on reduction flanges and on sealing flanges. Check O ring and sealing surfaces of the drop off plate.
	O ring is frozen due to contact with liquid nitrogen.	Immediately stop transferring liquid nitrogen. Warm up O ring with warm air! Wait until vacuum recovers and prevent liquid nitrogen from splashing on O rings.
	Pump stand is defective and does not reach the required vacuum of $10^{-6}$ mbar.	Check pump stand. Pumping directly against a sealing cap produces pressure below $10^{-6}$ mbar.

<b>Problem indicators</b>	<b>Possible reasons</b>	<b>Solutions</b>
The OVC becomes cold and wet.	Pumping was stopped. Vacuum is broken or worse than $10^{-3}$ mbar.	Do not remove the pumping unit until collection of liquid helium starts in the helium vessel! Find reason of vacuum problem (O ring, pump, frozen O ring) and proceed following the points mentioned there.
	Cold leak after transportation (transportation damage).	Contact Bruker/Spectrospin for further help.
Room temperature bore tube shows cold spot (condensing or freezing moisture on the bore tube).	Room temperature bore tube and N <sub>2</sub> bore are not aligned correctly and are touching each other.	Stop the cool down procedure. Warm up the system and repeat the alignment.
No liquid nitrogen is found when starting to pump back liquid nitrogen after pre-cooling.	The precooling L-tube is not inserted completely into the syphon.	Insert the L-tube completely into the syphon.
	The precooling L-tube is not tight in the syphon.	Check the lower end of the precooling L-tube for deformations and spread it slightly to fit into the syphon.
	No liquid nitrogen is collected into the helium vessel.	Check with the dipstick, to be sure that the helium vessel is completely empty of liquid nitrogen and of frozen nitrogen! (nitrogen ice).
The helium manifold becomes very cold and icy during pumping while pumping and flushing.	There is remaining liquid nitrogen in the helium vessel, boiling off strongly during pumping.	Stop pumping immediately and carefully remove all liquid nitrogen through the L-tube. Check with the dipstick to be sure that the helium vessel is completely empty of liquid nitrogen and of frozen nitrogen! (nitrogen ice).

Problem indicators	Possible reasons	Solutions
After some intervals of pumping and flushing it is not possible to reach a vacuum in the range of 1 mbar.	The spheres in the quench valves are not sitting correctly in the O rings and thus the quench valves are leaky.	Immediately stop pumping. Remove frozen air and frozen moisture with warm helium gas. Make tight the quench valves by slightly greasing the O rings and checking the position of the spheres. Check with the dipstick to be sure that the helium vessel is completely empty of liquid nitrogen and of frozen nitrogen! (nitrogen ice).
	There is remaining liquid nitrogen in the helium vessel, boiling off strongly during pumping.	Stop pumping immediately and carefully remove all liquid nitrogen through the L-tube. Check with the dipstick to be sure that the helium vessel is completely empty of liquid nitrogen and of frozen nitrogen! (nitrogen ice).
Frozen nitrogen (nitrogen ice) is found in the helium vessel.	Pumping intervals during pumping and flushing were too long and remaining nitrogen was boiling off and then freezing.	Warm up the magnet coil with warm helium gas through the L-tube and the syphon until the whole coil is warmer than 90° Kelvin. Check the temperature of the coil with the ACD*. Repeat pumping and flushing and carefully check with the dipstick to be sure that the helium vessel is completely empty of liquid nitrogen and of frozen nitrogen! (nitrogen ice).
The transfer of liquid helium does not start.	The transfer pressure in the transport dewar is too low.	Increase the transfer pressure.
	The transfer dewar is leaky, there is no transfer pressure built up.	Check transport dewar for leakage. Make all connections tight.



Problem indicators	Possible reasons	Solutions
	The transport dewar is empty.	Use new transport dewar.
	The syphon or the helium transfer line are blocked with ice.	Check syphon and transfer line for blockages, remove ice with warm helium gas.
The cool down of the magnet coil does not proceed although helium is transferred from the transport dewar into the cryostat.	The helium transfer line may be defective.	Check the helium transfer line for icing. If there are cold spots visible, replace the transfer line.
	The extension is not mounted on the transfer line. The liquid helium is not forced to flow through the syphon down to the bottom of the helium vessel.	Mount the extension piece on the transfer line. Check the helium transfer line to be inserted completely into the syphon.
The zero reading of the helium level meter can not be adjusted at the beginning of the cool down with liquid helium.	The level sensor is not connected properly with the connector in the helium manifold.	Check the connection in the helium turret between level sensor and connector.
	The level meter is defective.	Check the level meter with the 0% calibration plug.
The helium level never reaches 100% after the cool down.	The transport dewar is empty, no more helium is transferred into the cryostat.	Use new transport dewar.
	The levelsensor is disturbed by the transfer line's extension piece.	Stop transferring helium. Remove the transfer line and measure the helium level after some minutes without transfer line.
	The final filling should not be done through the syphon. The helium in the vessel is disturbed too much.	Use for final filling and for refilling of the cryostat the helium transfer line <b>without</b> extension piece.

<b>Problem indicators</b>	<b>Possible reasons</b>	<b>Solutions</b>
The OVC becomes wet and cold.	Pumping was stopped. Vacuum is broken or worse than $10^{-3}$ mbar.	Do not remove the pumping unit until collection of liquid helium starts in the helium vessel! Find reason of vacuum problem (O ring, pump, frozen O ring) and proceed following the points mentioned there.
	Cold leak after transportation. (transportation damage)	Contact Bruker/Spectrospin for further help.
Room temperature bore tube shows cold spot (condensing or freezing moisture on the bore tube).	Room temperature bore tube and N <sub>2</sub> bore are not aligned (correctly and are touching each other).	Stop the cool down procedure. Warm up the system and repeat the alignment.
After cool down the helium boil off is higher than specified. (up to 5 times)	Normal behaviour. The radiation shields and the insulation need some days to reach final low temperatures.	Wait some days and super- vise helium boil off. The presence of the current lead in the left helium turret during charging and shimming helps to cool down the radiation shield.
After cool down the nitrogen boil off is zero.	The nitrogen security flow system has not been mounted correctly during the cool down with helium. Due to supercooling of the nitrogen vessel during cool down with helium, air was sucked into the N <sub>2</sub> vessel and ice is blocking the N <sub>2</sub> turrets.	Immediately check the nitrogen turrets for the presence of ice and remove the ice with warm helium gas!

### 7.3 Trouble shooting during Charging and Shimming

Problem indicators	Possible reasons	Solutions
The current lead can not be inserted completely into the connector.	The connector is covered with ice. (frozen moisture or frozen nitrogen)	Carefully remove the ice with warm helium gas. To remove little ice spots use the dipstick as tubing for the warm helium gas.
	The shorting plug was not removed after cool down.	Remove the shorting plug with the extraction rod.
	The orientation of the current lead is not correct.	Turn the current lead carefully until it can be inserted correctly into the connector.
The shim heaters and/or the main heater can not be activated with the cryo power supply.	The current lead is not inserted correctly into the connector.	Turn the current lead carefully until it can be inserted correctly into the connector.
	The connector is covered with ice. (frozen moisture or frozen nitrogen)	Carefully remove the ice with warm helium gas. To remove little ice spots use the dipstick as tubing for the warm helium gas.
	The connectors "A" and "B" of the control cable are mismatched on the current lead's connectors.	Connect the control cable correctly to the current lead. See figures in chapter 3 for details of the current lead.
With closed main heater it is not possible to do the 100 A (120 A) test with the cryo power supply.	The main current cable is not connected properly to the current lead and/or the cryo power supply.	Connect the main current cable correctly to the current lead and the cryo power supply.
	The switch "Main Coil/OFF/Shim Coil" is not put on the "Main Coil" position.	Put the switch on the "Main Coil" position.

Problem indicators	Possible reasons	Solutions
With closed main heater it is not possible to do the 100 A (120 A) test with the cryo power supply.	The main current contacts on the current lead and/or the main current cable are oxidated and thus have too high resistance.	Clean main current contacts carefully. Connect current lead and main current cable correctly.
	The cryo power supply and/or the main current cable are defective.	Check the cryo power supply and the main current cable with a short circuit plug.
The sense voltage can not be set correctly to charge the magnet.	The main heater switch is set to the "OFF" position. The main switch is not opened.	Put the main heater switch to the "ON" position and check the main heater current to be adjusted correctly.
	The main heater current is set too low. The main switch is not opened.	Adjust main heater current correctly. See chapter 3.1 for specified values.
	The auxiliary shorting plug is inserted in the right helium turret and makes a short circuit across the main coil.	Remove the auxiliary shorting plug.
	600 MHz systems: The 22 pin shorting plug is inserted in the rear helium turret and makes a short circuit across the main coil.	Remove the 22 pin shorting plug.
The magnet quenches during charging.	Happens sometimes, caused by internal stress during charging.	Repeat cool down with helium within an hour after the quench. Wait the indicated time before charging the magnet again. For details see chapters 3 and 4 in the manual.
	The helium level was too low for charging.	Never try to charge the magnet with less than the minimum allowed level in the helium vessel. See chapter 3 for details.

Problem indicators	Possible reasons	Solutions
The magnet quenches during charging.	The cryo power supply is defective! The main current is oscillating.	Replace cryo power supply.
The main coil switch can not be closed on field.	The helium level is too low for charging. The main coil switch is not covered with liquid helium.	Never try to charge the magnet with less than the minimum allowed level in the helium vessel. See chapter 3 for details.
	The cryo power supply is defective! The main current is oscillating.	Replace cryo power supply.
The shim current can not be set correctly.	The control cable is not connected correctly to the current lead and/or the cryo power supply.	Correctly connect the control cable to the current lead and to the cryo power supply.
	The switch "Main Coil/OFF/Shim Coil" is not put on the "Shim Coil" position.	Put the switch "Main Coil/OFF/Shim Coil" on the "Shim Coil" position.
The shim current stops at approximately 80 mA and can not be set to a higher value	"A" and "B" connectors of the control cable are mismatched on the current lead. <b>Caution:</b> The shim current flows through the main heater and will open it!	Immediately check whether the magnet is in the persistent mode anymore. If not, immediately prepare the main current cable and put the cryo power supply in the "Main Coil" mode. Recharge the magnet and make it persistent on the correct field!
The shims have no effect on the NMR signal.	The shim heater current is set too low. The shim switches are not opened.	Set the shim heater current to the specified value according to chapter 3.

<b>Problem indicators</b>	<b>Possible reasons</b>	<b>Solutions</b>
The magnet can not be shimmed to reach specifications again.	There is some magnetic material in the room temperature bore tube (bostich, iron chip, rusty dust or similar).	Carefully clean the room temperature bore tube with a wet kleenex. <b>Caution:</b> Magnetic chips will be strongly attracted and will be drawn to the magnetic center. Try to wrap them up with the kleenex at the end of the bore tube!
	Massiv ferromagnetic parts are in the vicinity of the magnet that are strongly influencing the magnet.	Keep maximum possible distance between magnet and ferromagnetic parts. Repeat cryoshimming, starting with the low order shims and the values stated in chapter 3.1 of the manual.
The shimming procedure produces irreversible field shift.	Normal behaviour with 600 MHz systems.	Charge the magnet to a higher (lower) field as stated in chapter 3. The $Z^2$ -shift consists of the values of the $Z^2$ -shim itself and the effects of shimming.

## 7.4 Trouble shooting during Standard Operation

Problem indicators	Possible reasons	Solutions
The N <sub>2</sub> boil off falls to zero.	The N <sub>2</sub> neck tubes are blocked with ice.	Remove the heat exchangers and remove the frozen moisture. Check the O rings in the heat exchangers and mount them correctly on the N <sub>2</sub> turrets.
	During refill of helium supercooling of the N <sub>2</sub> vessel leads to reduced boil off.	Check some hours after the refill of helium, that there is normal N <sub>2</sub> boil off. Otherwise remove ice as described above.
	The N <sub>2</sub> vessel is empty.	Immediately refill the N <sub>2</sub> vessel. Keep a filling record. For specified hold time see chapter 4.
The helium boil off falls to zero.	The atmospheric pressure is increasing.	Normal behaviour, watch helium boil off daily.
	The He neck tubes are blocked with ice.	Immediately call a Bruker/Spectrospin service engineer. <b>Do not try to remove ice in the helium turrets without special knowledge!</b>
The helium boil off is too high.	The helium level meter is permanently on (service mode) or used frequently.	Measure the helium level once a week or less. Keep a helium level record. Every measuring of the helium level incorporates some helium losses due to the heating of the sensor.
	The atmospheric pressure is decreasing.	Normal behaviour. The Electronic Atmospheric Pressure Device (EAPD) is able to stabilize the pressure in the helium vessel within 0,1 mbar. (optional)

<b>Problem indicators</b>	<b>Possible reasons</b>	<b>Solutions</b>
The helium boil off is too high.	The pressure in the helium vessel is increasing due to blocked helium neck tubes or blocked helium manifold.	Immediately check helium boil off. If the helium neck tubes are blocked immediately call a Bruker/Spectrospin service engineer. <b>Do not try to remove ice in the helium turrets without special knowledge!</b>
	The radiation baffles are not inserted in the helium turrets.	Insert the radiation baffles into the left (and rear) helium turret.
	The helium oscillation damper is not mounted on the left helium neck tube.	Mount the helium oscillation damper on the left helium neck tube.
The NMR spectrum shows strong vibrations at approximately 43 Hz.	The helium oscillation damper is not mounted on the left helium neck tube.	Mount the helium oscillation damper on the left helium neck tube.



## 7.5 Trouble shooting during Discharging and Warm up

Problem indicators	Possible reasons	Solutions
The shorting plug cannot be removed from its place.	The connectors are covered with ice.	Carefully remove the ice with warm helium gas. To remove little ice spots use the dipstick as tubing for the warm helium gas.
The current lead can not be inserted completely into the connector.	The connector is covered with ice. (frozen moisture or frozen nitrogen)	Carefully remove the ice with warm helium gas. To remove little ice spots use the dipstick as tubing for the warm helium gas.
	The shorting plug was not removed.	Remove the shorting plug with the extraction rod.
	The orientation of the current lead is not correct.	Turn the current lead carefully until it can be inserted correctly into the connector.
The shim heaters and/or the main heater can not be activated with the cryo power supply.	The current lead is not inserted correctly into the connector.	Turn the current lead carefully until it can be inserted correctly into the connector.
	The connector is covered with ice. (frozen moisture or frozen nitrogen)	Carefully remove the ice with warm helium gas. To remove little ice spots use the dipstick as tubing for the warm helium gas.
	The connectors "A" and "B" of the control cable are mismatched on the current lead's connectors.	Connect the control cable correctly to the current lead. See figures in chapter 3 for details of the current lead.
With closed main heater it is not possible to do the 100 A (120 A) test with the cryo power supply.	The main current cable is not connected properly to the current lead and/or the cryo power supply.	Connect the main current cable correctly to the current lead and the cryo power supply.

<b>Problem indicators</b>	<b>Possible reasons</b>	<b>Solutions</b>
With closed main heater it is not possible to do the 100 A (120 A) test with the cryo power supply.	The switch "Main Coil/OFF/Shim Coil" is not put on the "Main Coil" position.	Put the switch on the "Main Coil" position.
	The main current contacts on the current lead and/or the main current cable are oxidated and thus have too high resistance.	Clean main current contacts carefully. Connect current lead and main current cable correctly.
	The cryo power supply and/or the main current cable are defective.	Check the cryo power supply and the main current cable with a short circuit plug.
The sense voltage can not be set correctly to charge the magnet.	The main heater switch is set to the "OFF" position. The main switch is not opened.	Put the main heater switch to the "ON" position and check the main heater current to be adjusted correctly.
	The main heater current is set too low. The main switch is not opened.	Adjust main heater current correctly. See chapter 3.1 for specified values.
	The auxiliary shorting plug is inserted in the right helium turret and makes a short circuit across the main coil.	Remove the auxiliary shorting plug.
	600 MHz systems: The 22 pin shorting plug is inserted in the rear helium turret and makes a short circuit across the main coil.	Remove the 22 pin shorting plug.
The magnet quenches during charging.	The helium level was too low for charging.	Never try to charge the magnet with less than the minimum allowed level in the helium vessel. See chapter 3 for details.
	The cryo power supply is defective! The main current is oscillating.	Replace cryo power supply.

<b>Problem indicators</b>	<b>Possible reasons</b>	<b>Solutions</b>
The shim current can not be set correctly.	The control cable is not connected correctly to the current lead and/or the cryo power supply.	Correctly connect the control cable to the current lead and to the cryo power supply.
	The switch "Main Coil/OFF/Shim Coil" is not put on the "Shim Coil" position.	Put the switch "Main Coil/OFF/Shim Coil" on the "Shim Coil" position.
The nitrogen boil off is zero after the discharging of the magnet.	Due to supercooling of nitrogen air was sucked into the nitrogen vessel and frozen moisture blocks the nitrogen turrets.	Remove the ice from the nitrogen turrets.
After breaking the vacuum with nitrogen with a needle valve, the helium and nitrogen boil off are very high.	The liquid cryogenes have not been removed from the vessels before breaking the vacuum.	Blow out the liquid helium with warm helium gas blown through the L-tube into the syphon. Blow out the liquid nitrogen into a transport dewar.
The vacuum is not broken completely after 12 hours.	The vacuum valve has closed itself due to pressure differences.	Block the operator of the vacuum valve with the split plastic tube.
The room temperature bore tube is wet and cold before disassembly of the dewar.	The magnet coil has not yet warmed up completely.	<b>Wait one more day.</b> Never open a dewar before the room temperature bore tube is warm and dry!

## 7.6 Trouble shooting of the Anti Vibration Magnet Stand

Problem indicators	Possible reasons	Solutions
The anti vibration magnet stand does not reach the operating position.	The air controller is on the "DOWN" position.	Put the air controller to the "UP" position.
	The pressure of the gas supply is too low.	Check the pressure of the gas supply. It must be in the range of 5 bar to 8 bar (70 psi to 112 psi).
	The pneumatic tubings or connectors are leaky.	Check the pneumatic tubings and connectors for leakage.
	The NMR magnet system is not leveled correctly.	Deactivate the anti vibration units or the vibration dampers. Check the leveling of the cryostat as described in chapter 6.
	The operating position is not leveled correctly.	Activate the anti vibration units or the vibration dampers. Check the leveling of the cryostat as described in chapter 6. Contact a Bruker/Spectrospin service engineer to repeat the leveling of the NMR magnet system if necessary.
	A level valve is defective.	Contact a Bruker/Spectrospin service engineer to exchange the defective level valve..
	The membrane of a vibration damping unit is defective.	Contact a Bruker/Spectrospin service engineer to repair or exchange the defective vibration damping unit.
The NMR spectrum shows massive vibrations.	The air controller is on the "DOWN" position.	Put the air controller to the "UP" position.

<b>Problem indicators</b>	<b>Possible reasons</b>	<b>Solutions</b>
	The vibration dampers are pumped to strongly. The damping efficiency is reduced.	Check the pressure in the vibration dampers. Adjust the pressure as described in chapter 6.
	The cryostat can not play freely in the operating position. The dewar has direct mechanical contact with the floor.	Check the leveling of the NMR magnet system with deactivated as well as with activated vibration damping units. With activated vibration damping units no scratching or touching between cryostat and magnet stand is observed.
	The dewar has direct mechanical contact with the floor.	Check that no direct mechanical contact between cryostat and floor is produced by any accessory close to the NMR magnet system.

