

Bruker BioSpin

NMR CASE •

Installation Manual

Version 011

NMR Spectroscopy

think forward

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Introduction

Warnings 1.1

WARNING!



The NMR CASE has to be installed on top of a NMR magnet cryostat.

DANGER!



This magnet creates a very STRONG MAGNETIC FIELD.

Objects and tools can be attracted to the magnet with great force and may cause damage.

Use only NON-MAGNETIC tools and equipment.

A non-magnetic TOOL SET is supplied with the NMR Case.

Read and follow carefully this installation manual. Product description, usage, scope of manual, etc.

Disclaimer 1.2

BRUKER accepts NO responsibility for damages resulting from:

- 1. Installations by untrained personnel.
- 2. Not following the manuals provided.

System Requirements

1.3

Before installing the NMR Case, read and follow the System Requirement manual P/N B2928.

Non-magnetic Tool Set P/N B2852

1.4

Parts list:

- One non-magnetic Philips screwdriver #2, P/N 92437
- One collar, mounted to the screwdriver, P/N B2817
- One non-magnetic wrench 10 mm, P/N B2438
- One circular level, P/N 92438.

If you cannot find this non-magnetic tool set, call BRUKER and order one (The Bruker part number is B2852).

WARNING!



To avoid damage to the magnet cryostat, use only NON - MAGNETIC tools.

Non-magnetic Ladder

1.5

To install the NMR case on top of the magnet cryostat, a non-magnetic ladder made from wood or aluminum must be available. Bruker does not supply non-magnetic ladders.

WARNING!



To avoid damage to the magnet cryostat, use only $\ensuremath{\mathsf{NON}}$ - $\ensuremath{\mathsf{MAG-NETIC}}$ equipment.

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Parts List 2

NMR CASE Parts List 2.1

One NMR Case base assembly	
One gray security ring (mounted to base)	
Two sets of telescopic front legs	
One (or several) exchangeable sample holder trays	P/N B2436
One BST adapter collar (mounted to base)	P/N B2383
One motion controller unit	P/N B3165
Two mounting brackets	P/N B3015
One power cord	
 One spare fuse (inside the fuse holder) 	
One pneumatic hose Ø 4 mm	
One pneumatic cable assembly	
One non-magnetic tool set	P/N B2874
One pneumatic hose Ø 6 mm	
One adapter Ø 6 to Ø 8 mm	
• One T piece Ø 8 mm	
One BSMS sample control modifier cable	P/N B1428
One "System requirement" manual	P/N B2928
One "Trouble shooting guide"	P/N B2927
 One instruction sheet "Interface tube for new BST" 	P/N B3696
This manual	P/N B2926
One instructions for modifier cable	P/N B2300

Parts List

Installation Instructions

Short Installation Description

3.1

- Mount the front legs to the NMR Case base assembly.
- Mount the base assembly to the shim upper stack.
- Align the base to the magnet cryostat.
- Adjust the length of both telescopic front legs and level the base.
- Connect the pneumatic hoses.
- Install the motion controller unit
- Switch on the motion controller.
- Install a sample holder tray.
- First tests.
- Operation.

Refer to Appendix <u>"Illustrations" on page 19</u> for figures and illustrations supporting the installation and operation.

Mount the Front Legs to the Base

3.2

Locate the two sets of telescopic front legs. Mount the legs using the plastic hex screws. Adjust the legs into the shortest position. Tighten all eight plastic hex screws with the non-magnetic wrench.

Mount the Base to the Shim Stack

3.3

The shim stack has to have a specific orientation. Read and follow <u>section 1.5</u> of the "System requirement manual" P/N B2928.

The interface tube has to be selected to match the BST. Read and follow the instruction sheet "Interface tube for new BST" P/N B3696.

- 1. On the base plate, locate the adapter collar and remove the warning label from the gray safety ring.
- 2. Rotate the gray safety ring until the three screws for the collar are visible through the openings.

Installation Instructions

- Using the non-magnetic Philips screwdriver, loosen these three screws, but do not remove them.
- 4. Check that the collar can now be rotated.
- 5. Use a non-magnetic ladder and remove all labels from the shim stack top head. Refasten the labels to the lower, narrow part of the shim stack.
- 6. Lift up the NMR Case base and line up the openings of the adapter collar with the corresponding nipples of the shim stack.
- 7. Push the collar down over the top head of the shim stack. It should sit firmly and one should feel the engagement of the O-ring inside the adapter collar with the shim stack top head. See *Figure A.3.* to *Figure A.5.* for details.
- 8. Using the non-magnetic wrench, loosen the white hex screws for the front legs and pull the legs down until they rest on the magnet cryostat. Rotate the lower leg piece until it is lined up with the surface of the magnet cryostat.
- 9. Rotate the NMR Case base plate until there is equal distances from both nitrogen (and helium) towers.
- 10. Place the circular level (from the tools set) on top of the NMR Case base plate.
- 11. Observing the bubble of the circular level, adjust the length of both front legs until the base plate is level. The bubble must be inside the innermost ring. Tighten all white hex screws of the front legs with the non-magnetic wrench.
- 12. Using the non-magnetic Phillips screwdriver, tighten all three screws on the adapter collar.
- 13. Check the circular level. If necessary, readjust the length of the front legs until the base plate is level.
- 14. Unscrew the screw for the base cover and lift up the base cover slightly.
- 15. Remove the gray safety ring. Check that the seal ring and the gasket are still in place.
- 16. Place back the base cover and fasten the screw.

Connect the Pneumatic Hose Assembly

3.4

The sample lift air hose going to the shim stack needs to be rerouted through the NMR Case.

Remove the sample lift air hose (white color) from the shim stack and push it into the corresponding connector located on the manifold below the NMR Case base plate. With a short piece of 8-mm hose, connect the outlet on the other side of the manifold back to the shim stack sample lift air inlet.

Connect the pneumatic hose assembly to the NMR Case manifold. The hoses are labeled in sequence # 5, 1, 2, 3, 4, 6, 7. Push the hoses # 5, 1, 2, 3, 4, 6 all the way into the pneumatic connectors located on the manifold below the NMR Case base plate. Hose # 7 goes into the pneumatic connector located at the adapter collar.

3.5

Placement 3.5.1

Place the motion controller unit on top of a rack or some other convenient place. It must be at least 2 meters (6 feet) away from the magnet cryostat dewar. The environment temperature range must be 0 °C to + 50 °C.

It can also be mounted inside the NMR console. For console mounting, remove the black rails of the motion controller and mount two flat mounting brackets instead (B3015). Remove the back panel of the NMR console and mount the motion controller in a free spot (usually in the top left corner).

WARNING!



The motion controller unit is magnetic and does not work in the magnetic field.

It can be attracted to the magnet with great force.

It must be 2 meters (6 feet) away from the magnet cryostat dewar.

Main Air Connection 3.5.2

Connect the motion controller inlet marked "MAIN AIR" to a compressed air supply (for specifications, see the manual "System Requirements P/N B2928, <u>section 1.7</u>). To make this connection, use a length of 6 mm hose, a 6 to 8 mm adapter and a Tee piece (all supplied with the NMR Case).

Power 3.5.3

Using the power cable, connect the inlet marked "110/220 VAC " to a power source, either 110 or 220 V and 50 or 60 Hz. The unit selects the voltage automatically, there is no voltage selector switch.

Pneumatic Hose Assembly

3.5.4

Connect the pneumatic hose assembly to the motion controller as follows:

Controller I/O	Hose label	Hose	NMR Case
CYL A, L-IN	L-IN	# 1	Manifold # 1
CYL A, L-OUT	L-OUT	# 2	Manifold # 2
CYL B, EXT	EXTEND	# 3	Manifold # 3

CYL B, RET	RETRACT	# 4	Manifold # 4
SENSOR 1	SENSOR	# 5	Manifold # 5
SL-SENSE	SL-SENS	# 6	Manifold # 6
SENSOR 2	SENSOR 2	# 7	Adapter Collar

Test Connector (9 pins)

3.5.5

Do not connect anything to the nine pin connector. It is for test purposes only.

Switch ON the Motion Controller

3.6

At this time the motion controller can be turned on.

Observe the black push lever on the side of the case body. It should move to the right and come out all the way. Test that the push lever is powered by air by grabbing it hand and trying to move it to the left and in. There should be a firm resistance.

WARNING!



Leave the motion controller ON at all times during operation.

Switch it off only for test purposes or if it is not used anymore.

Press the reset button.

This clears the sample tracking memory of the controller.

Mount the Sample Holder Tray

3.7

For the first test, leave the sample holder tray empty without samples.

- 1. Locate the sample holder tray.
- 2. Make sure that all samples are removed from the tray.
- 3. Locate the green "START" position.
- 4. While pushing the tray lock lever inside the tray to the position "Unlock", rotate the sample stop head to the location of the "START" position.
- 5. Let go the tray lock lever. Check that it snaps back to the locked position.
- 6. Grab and lift the sample holder tray at the handle.
- 7. Line up and lower the sample holder tray over the NMR Case base cover.

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- 8. Line up the sample stop head with the black off-center pin and engage it.
- 9. Line up the center with the black on-center pin and engage it.
- 10. Engage the tray into the side pin.
- 11. The sample holder tray settles down. Make sure that it seated correctly all the way down.
- 12. Check that the tray lock lever is in the unlocked position.

First Manual Test Without Samples

3.8

- Manually switch the sample lift ON.
- 2. After about 6 seconds the sample holder tray should rotate away from position 0 and settle on position 1.
- 3. Manually switch OFF the sample lift.
- 4. Wait until the sample lift airflow is completely OFF (no noise anymore), then switch it on again.
- 5. The NMR Case motion controller should advance (rotate) the sample holder tray to the next sample position.
- 6. This sequence can be continued as necessary by switching the sample lift airflow manually OFF and ON.

If this test is NOT successful, then read and follow the "NMR Case trouble shooting guide", P/N B2927



Important: The sample holder tray advancing movement needs to be adjusted. Read and follow **section 1.2.5 and 6** of the troubleshooting guide.

First Manual Test With Samples

3.9

For the first tests, use inexpensive and empty sample tubes only.

- 1. Place two (or more) spinners with empty sample tubes into the next available positions of the sample holder tray on top of the magnet.
- 2. Manually switch the sample lift ON.
- The NMR Case motion controller should sense the sample lift (after 6 seconds) and advance (rotate) the sample holder tray to the next sample position containing the first sample.
- 4. For about 2 seconds the sample tube should float on the sample lift air, before it is inserted rapidly (1 sec) down into the BST shim stack. There it should float again.

Installation Instructions

- 5. Manually switch the sample lift OFF.
- 6. With the decreasing sample lift air-flow, the sample should slowly go down all the way into the shim coil.
- 7. The NMR case motion controller should sense the absence of sample lift airflow and retract the advance lever (but not rotate the sample holder tray yet).
- 8. Manually switch the sample lift ON.
- 9. With the increasing sample lift airflow, the sample should come up all the way into the sample holder tray within 10 seconds. If it does not come up all the way or if it takes longer than 10 seconds, the sample lift airflow must be increased. Read and follow section 1.1 and 1.3 of the troubleshooting guide.
- 10. The motion controller should sense the sample top position and advance (rotate) the sample holder tray to the next position.
- 11. The next sample should be inserted down into the shim stack and float on the sample lift air.
- 12. This sequence can be continued as necessary by switching the sample lift airflow manually OFF and ON.

If this test is NOT successful, then read and follow the "NMR Case trouble shooting guide", P/N B2927

Adjustments for Reliable Long Term Operation

3.10

The following 3 items need to be properly adjusted for a reliable long-term operation of the NMR Case. Read and follow the "NMR Case troubleshooting guide", P/N B2927, as indicated:

Item	Necessary Adjustments	Troubleshooting Guide
1	Level of the NMR Case	Section 2.1
2	Sample lift air flow	Section 1.1 and Section 2.4
3	Sample holder tray movement	Section 2.5 and Section 2.6

Automatic Operation

4.1

- 1. If the manual test is successful, then the NMR Case is ready for automatic operation.
- 2. Read and follow the manual for the automation software: "How to set up Icon NMR' P/N B0951.
- 3. The NMR Case sample tray does not need to be filled sequentially with samples. There can be one or several empty positions between samples. The NMR Case will "jump" over these empty positions. However it does not jump on it's own, the sample lift needs to be switched ON and OFF the usual way and the sample tray will then advance to the next position. This feature is useful when measuring several small batches of samples, adding them to the already loaded sample holder tray without removing the tray from the magnet.
- 4. The NMR Case will stop advancing when the sample lift is not activated anymore.
- 5. The NMR Case will remember if a sample is still inside the shim coil and has not been ejected yet. If this sample is being removed "manually" (by removing the sample tray, activating the sample lift manually and removing the sample by hand), then the sample holder tray will not advance anymore. To solve this situation, either insert another sample manually, or press the reset switch located at the right side of the motion controller. Switching off the power to the motion controller unit will NOT erase the sample tracking memory.
- 6. If a sample jams and does not get inserted all the way into the shim coil, then the sample changer does not advance the sample holder tray. Therefore the next sample lift sample insert sequence will be done with the same sample, until this sample it is successfully inserted. The software will not track this repetition; therefore some samples at the end of the queue will not be measured.
- 7. Built in safety features will prevent sample damage in case of power and air failures.

Manual Operation

4.2

- The NMR Case can be used for manual operation by simply removing the sample holder tray and inserting the sample through the adapter collar into the shim stack. It is not necessary to remove the NMR Case base assembly and it is not necessary to switch off the motion controller.
- 2. In addition, it is possible to fill the sample holder tray with just one sample and then operating the sample lift manually in the usual way, without the automation software.

- 3. To use the NMR Case in automatic mode again, the sample tracking memory has to be cleared. While the sample lift is OFF, press the RESET button on the side of the motion controller.
- 4. Note: pressing the RESET button while the sample lift is ON does not work and the sample holder tray will not advance away from the position 0.

Second Sample Holder Tray

4.3

A second (or more) sample holder tray can be purchased under part number B2436 and be used to load fresh samples while the first tray is in use on top of the magnet. A simple swap of the trays keeps the spectrometer running, while the first tray can be conveniently loaded with new samples again.

1. Grab the sample holder tray at the handle and lift it straight up and away from the NMR Case base.

WARNING!



Do not swing the sample holder tray.

Watch out for the glass tubes close to the wrist. handle with care.

- 2. Place the sample holder tray on a secure table surface.
- 3. Remove all samples from the tray.
- 4. Locate the green "START" position.
- 5. While pushing the tray lock lever inside the tray to the position "Unlock", rotate the sample stop head to the location of the "START" position.
- 6. Let go the tray lock lever. Check that it snaps back to the locked position.
- 7. Insert a sample tube mounted in a spinner into sample position "1".
- 8. Insert more samples in sequential order. Any number of samples (up to 24) can be used.
- 9. Grab and lift up the sample holder tray at the handle.
- 10. Line up and lower the sample holder tray over the NMR Case base cover.
- 11. Line up the sample stop head with the black off-center pin and engage it.
- 12. Line up the center with the black on-center pin and engage it.
- 13. Engage the tray into the side pin.
- 14. The sample holder tray settles down. Make sure that it seated correctly all the way down
- 15. Check that the tray lock lever is in the unlocked position.
- 16. If accidentally there is a sample left inside the shim coil, it will be inserted into the start position of the sample holder tray.

Sample Diameters 4.4

The NMR Case can handle sample tubes with diameters from 2.5 mm up to 10 mm.

WARNING!



Do not use sample tubes with diameters larger than 10 mm, the glass tubes may break.

The sample lift airflow needs to be adjusted depending on the weight of the sample and the spinner. Heavier sample / spinners need a larger sample lift airflow. See the trouble shooting guide **Section 1.1** and **Section 2.4** on how to adjust the sample lift airflow.

For reliable long-term operation it is not recommended to mix lightweight and heavy sample /spinners in one sample holder tray.

Sample Diameter	Spinner Material	Spinner Color	Approved for NMR Case	Max Temp	Weight
5+2.5 mm	POM	Blue	Yes	50 °C	lightweight
8 mm	POM	Blue	Yes	50 °C	lightweight
10 mm	POM	Blue	Yes	50 °C	Medium weight
15 mm	PPO		NO	Do not use	
5+2.5 mm	PCTFE	Gray	Yes	80 °C	Medium weight
8 mm	PCTFE	Gray	Yes	80 °C	Medium weight
10 mm	PCTFE	Gray	Yes	80 °C	Medium weight
15 mm	PCTFE	Gray	NO	Do not use	
5+2.5 mm	Ceramic	White	Yes	80 °C	heavy
8 mm	Ceramic	White	Yes	80 °C	heavy
10 mm	Ceramic	White	Yes	80 °C	heavy
15 mm	Ceramic	White	NO	Do not use	

Temperature Range

4.5

The NMR Case sample changer can handle a temperature range of -20 $^{\circ}$ C to + 50 $^{\circ}$ C.

Operation

However it is acceptable to measure the NMR samples inside the probe with a temperature range of -50 °C to + 80 °C, because the sample lift air flow will warm up or cool down the sample and the spinner by the time they reach the NMR Case sample holder tray.

But a larger temperature range is not possible even using the higher rated white ceramic spinners, because the material of the NMR Case will get distorted by the hot shim upper stack and the hot spinners (Ceramic spinners hold the temperature for a long time).

The motion controller itself has a temperature range of 0 °C to + 50 °C.

Troubleshooting 4.6

For all troubleshooting issues, read and follow the "NMR Case Troubleshooting Guide", P/N B2927.

Illustrations



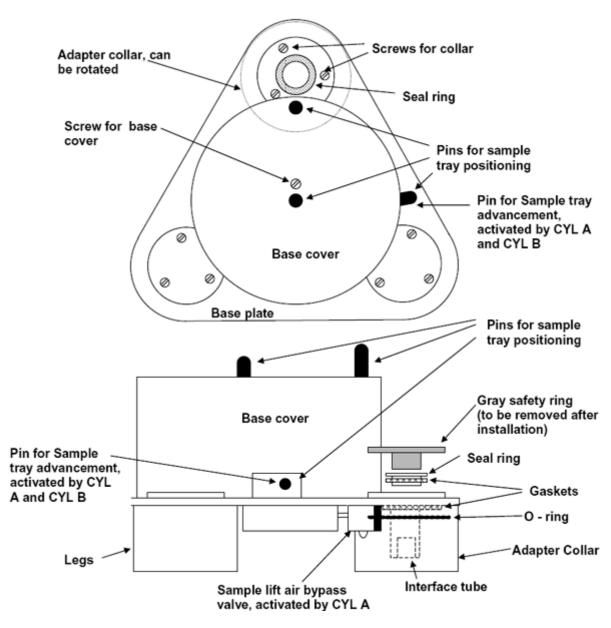


Figure A.1. NMR CASE Base

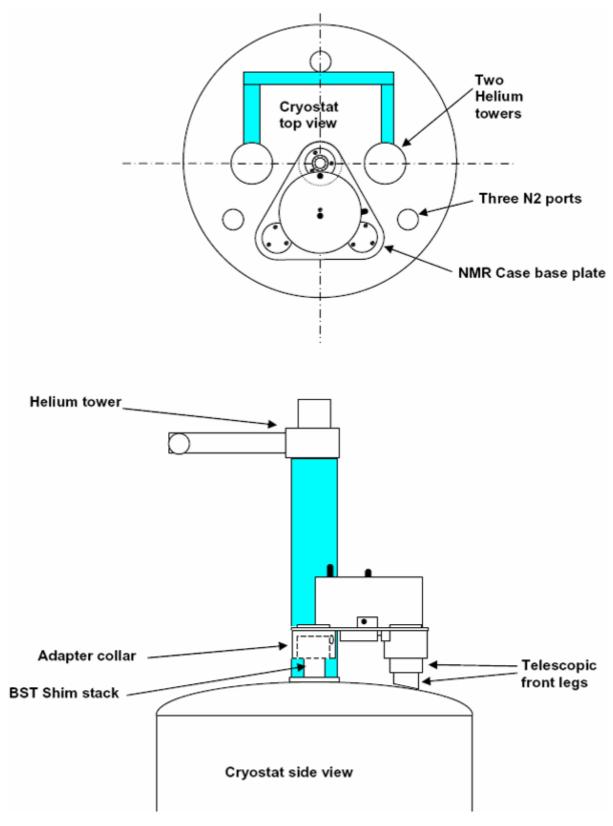


Figure A.2. NMR CASE Base Mounted

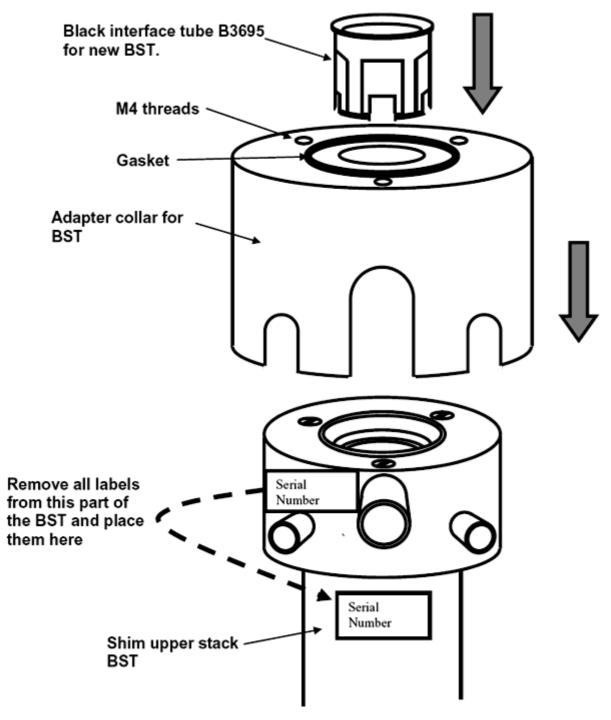


Figure A.3. Adapter Collar for the BST

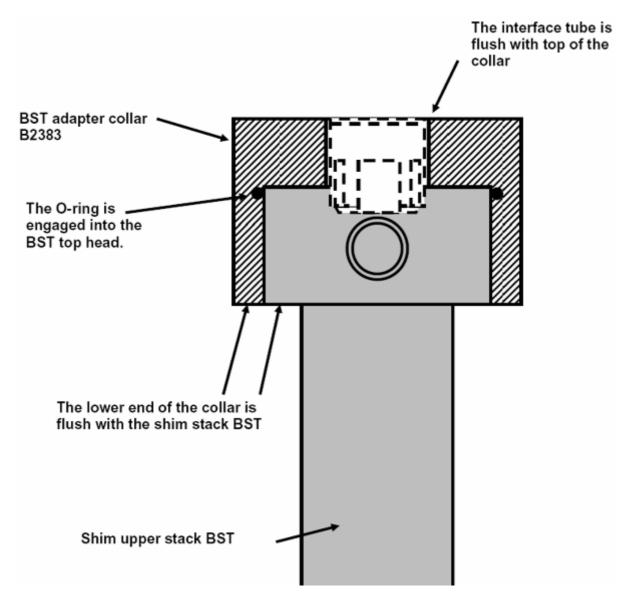


Figure A.4. Adapter Collar Installation

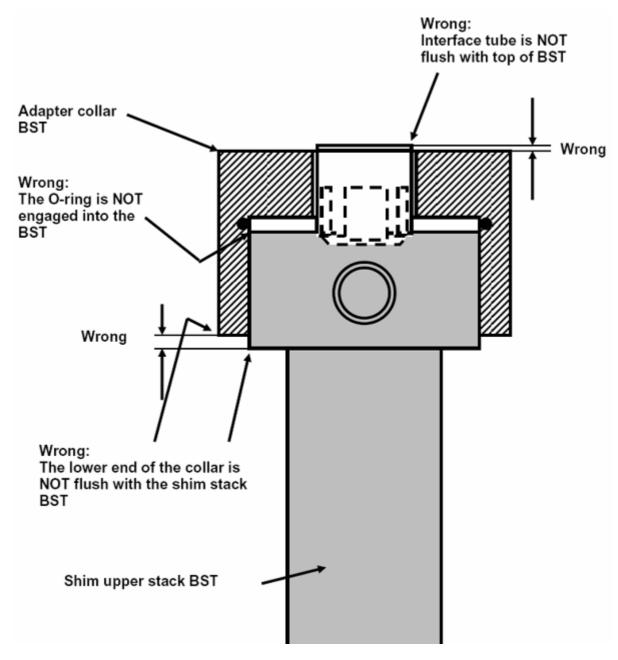


Figure A.5. Incorrect Installation of the BST Collar

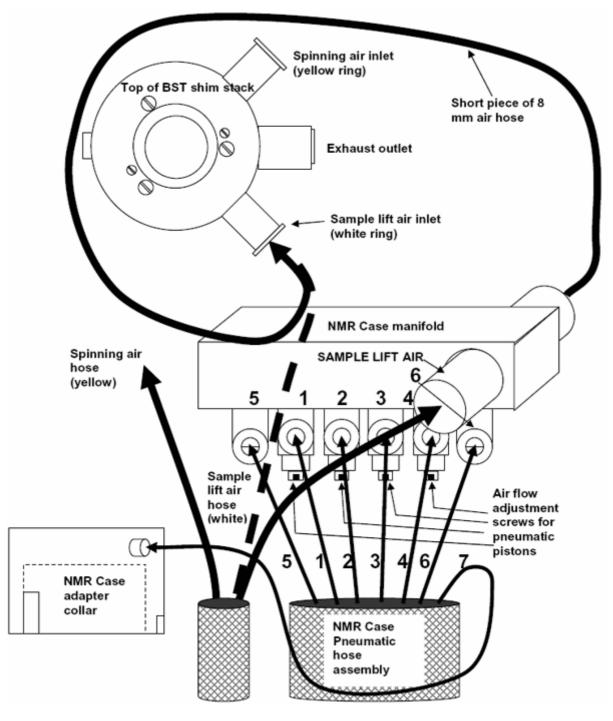


Figure A.6. Pneumatic Hose Connections

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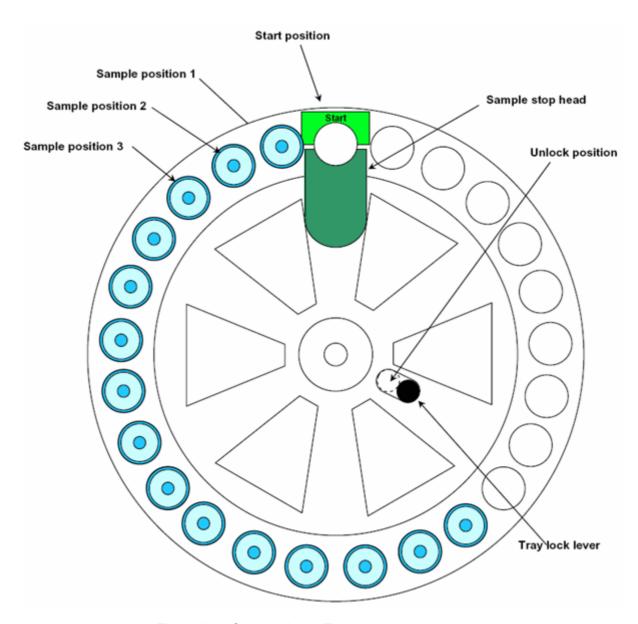


Figure A.7. Sample Holder Tray

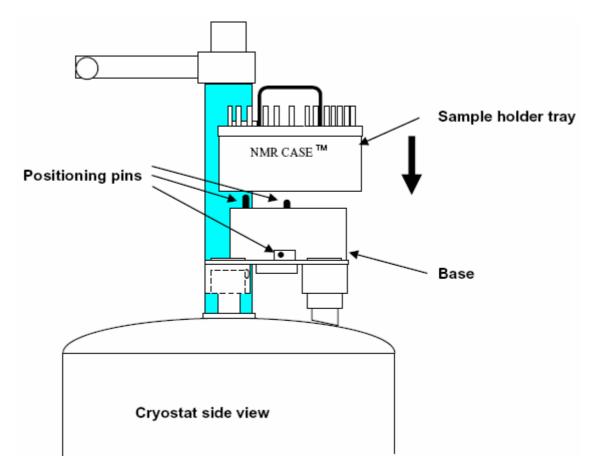


Figure A.8. Sample Holder Tray Line Up

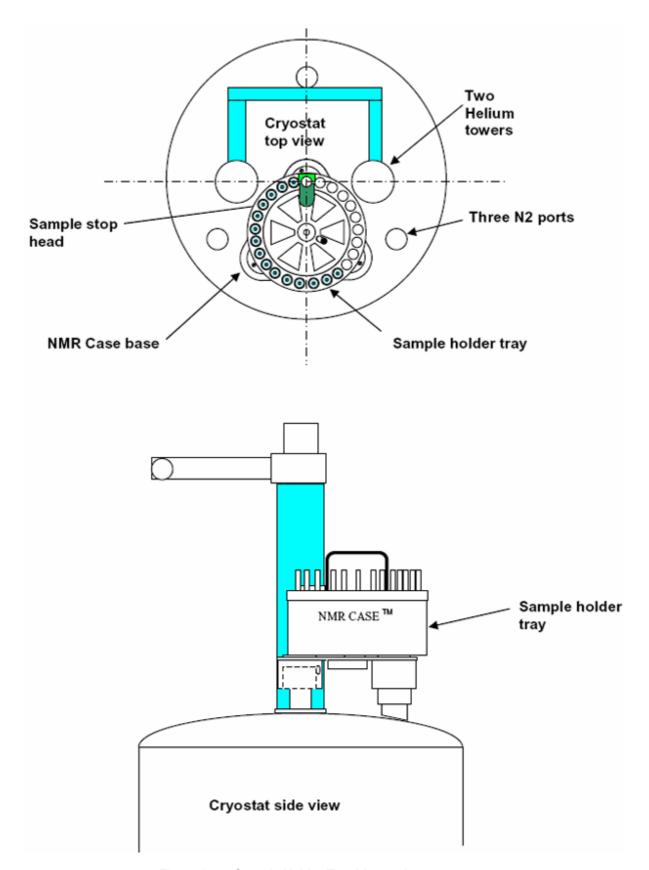


Figure A.9. Sample Holder Tray Mounted

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