

Bruker BioSpin



Troubleshooting Guide

Version 021

think forward

NMR Spectroscopy

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Contents

	Contents 3
1	Installation Problems5
1.1	Sample Eject (Lift) Problems5
	Sample lift air flow5
	Sample holder tray slamming5
	Light barrier
	Seals6
	Seating of the adapter collar6
	Alignment of the adapter collar, seal ring and brown interface tube6
	Sample lift air bypass6
	Interface tube inside the adapter collar
1.2	Sample Insert (Down) Problems
	NMR CASE or magnet not level
	BST shim stack with sharp edges
	Sample lift air bypass
	Too much sample lift air flow
	Sample holder trav does not advances firmly
	Alignment of the adapter collar seal ring and brown interface tube 8
	Interface tube inside the adapter collar
13	Sample holder trav does not advance at all
14	The sample holder tray advances when the eject air is turned OFF
1.5	The motion controller does not work
1.6	The sample holder tray slams samples when advancing 12
1.0	The adapter collar does not fit to the shim stack
1.8	The NMR CASE does not fit
1.0	The sample spin rate goes up to 150 Hz
1 10	Intermittent problems
1 11	Sample lift (eject) sometimes hangs on top of the BST and does NOT go
	up into the NMR Case
1 1 2	Sample lift (eject) always bangs on top of the BST and does NOT go up
1.12	into the NMR Case
1 13	Sample lift (eject) bangs just below the top of the sample holder trav 14
1.13	Sample insert (down) sometimes bands on top of the sample holder tray
1.14	
1 15	Sample insert (down) cometimes hangs at the white ring on top of the
1.15	adapter collar
1 16	The comple helder tray does not advance away from position 0.
1.10	Sometimes the tray rotates in the wrong direction
1.17	Sometimes the tray does not rotate
1.10	Sometimes and a stuck in brown interface type /incide the edenter set
1.19	Sometimes samples stuck in brown interface tube (inside the adapter col-
	ıar)

2	Problems During Operation17
2.1	The air cylinder is too weak and does not rotate the sample tray complete-
	ly 17
2.2	The air cylinder leaks oil
2.3	The NMR Case shares off sample tubes 18
2.4	The air cylinders do not work
2.5	The adapter collar cannot be removed from the BST upper stack 19
2.6	The black push lever on the side of the NMR Case feels loose and does
	not move at all
Α	Illustrations21
	Figures

Installation **Problems**

Troubleshooting Guide Version 021

Sample Eject (Lift) Problems

Sample lift air flow

When ejecting the sample, it must come up with speed (within 10 seconds) and climb all the way to the top of the NMR Case. There must be enough airflow so that the top of the spinner is pressed against the sample stop head on top of the sample tray. A setting of 850 for the sample lift air flow is the normal value for the operation with a NMR Case.

If the sample creeps up very slowly and barely reaches the sample stop, then the sample lift airflow must be increased. There is a dedicated method on how to adjust the sample lift airflow within the NMR console. It is done either by software or with the BSMS. Do not increase the console main air pressure. Read the corresponding manual on how to adjust the sample lift airflow (or ask Bruker for assistance).

In case the sample lift airflow needs to be increased fully to its maximum, then there is possible an air leak. Read and follow sections 1.1.3 to 1.1.8 to find the fault.

Note that the factory sample lift air flow setting is 750 and that the console will revert back to this factory setting after a power loss, unless the larger value of 850 is stored in memory.

Sample holder tray slamming

If the sample holder tray slams when advancing, it may need more sample lift air than necessary. See section 6 for adjustments.

Light barrier

There is a light barrier built into the top part of the BST shim stack, it slows down the sample lift. Use the sample control modifier cable P/N B1428 to disengage the light barrier. Read the instruction sheet P/N B2300.



1.1.1

1.1

1.1.2

1.1.3

1.1.5

To avoid sample lift air leaks, the following four seals must be properly installed (see Installation manual *Figure 1, page 19*):

- 1. Inside the adapter collar: one O-Ring.
- 2. On top of the adapter collar: one flat rubber seal.
- 3. Inside the large white round ring plate: one flat rubber seal.
- 4. One white or brown seal ring on top of the large white round ring plate.

Seating of the adapter collar

Check that the adapter collar is seated correctly on top of the BST shim stack. The lower end of the collar must be flush with the lower end of the top large portion of the BST.

Alignment of the	adapter collar, seal	ring and brown interface tube	1.1.6
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The adapter collar, seal ring and brown interface tube inside the collar must be aligned. Loosen the three screws of the collar and insert the gray safety ring through the seal ring into the brown interface tube. The safety ring serves as alignment tool. Tighten the three screws again and remove the safety ring.

Sample lift air bypass

There is a small pneumatic cylinder located inside aluminum housing underneath the NMR Case base plate (see *Figure A.2.* and *Figure A.3.*). This cylinder opens and closes a sample lift air bypass exit hole, located in the adapter collar. If the bypass exit hole is not closed completely, the sample will not eject to the top of the sample tray, but will sit on top of the BST instead.

Read section <u>1.1.8</u> on how to check for correct operation.

Interface tube inside the adapter collar

Inside the adapter collar is a tube, which interfaces the adapter collar with the BST shim upper stack (see *Figure A.2.* and *Figure A.3.* at the end of this manual and *Figure 3* at the end of the installation manual). The correct seating of the adapter collar and the interface tube is critical for the reliability of the sample changer.

To check the seating, first remove the 3 screws and then remove the NMR Case base plate, leaving only the collar in place.

Check that the bottom of the collar is flush with the BST. Check that the interface tube is flush with the top of the collar.

Check that the five fingers of the interface tube are not bent inwards and block the inside diameter of the BST.

1.1.7

1.1.8

See *Figure 4* at the end of the installation manual for the correct seating. See *Fig-ure 5* at the end of the installation manual for the incorrect seating.

If the collar cannot be seated correctly, then check the BST top head for parts sticking out (labels or covers).

If the O-ring does not engage onto the BST (it feels too tight), then apply some silicone grease onto the O-ring inside the collar.

If the interface tube is not flush with the top of the collar, then it may be an old version (made from brown plastic). Replace it with a new version made from black metal (P/N B3695).

If the inner diameter of the interface tube seems to be too small, then it may be an old version, made from brown plastic. Replace it with a new version made from black metal (P/N B3695).

Sample Insert (Down) Problems	1.2

NMR CASE or magnet not level

The shim stack must be vertical and the NMR Case horizontal aligned. Check it with the circular level (supplied with the tool kit). The bubble must be inside the innermost ring. Adjust the magnet stand and the telescopic front feet as necessary.

BST shim stack with sharp edges	1.2.2
---------------------------------	-------

On top of the BST, where the samples are inserted, there are some cutouts located on the inside diameter. Some cutouts have sharp edges. If this is the case (check it with a finger), either replace the BST or remove the sharp edges with a file.

Sample lift air bypass	1.2.3
------------------------	-------

After changing a sample, the new sample should stay on top of the sample holder tray for a short moment, before it is rapidly inserted into the BST (within 2 seconds).

There is a small pneumatic cylinder located inside aluminum housing underneath the NMR Case base plate (see *Figure A.2.* and *Figure A.3.*). This cylinder opens and closes a sample lift air bypass exit hole, located in the adapter collar. Check for the correct opening and closing of this bypass exit hole.

If the bypass exit hole is not open completely, the sample does not get inserted fast enough into the BST and may get stuck on top of the BST.

If the bypass exit hole is not closed completely, the sample will not eject to the top of the sample tray, but will sit on top of the BST instead.

If the bypass does not open or close, adjust the air flow valves. Refer to *Figure A.3.* at the end of this manual and locate the air flow adjustment screws number 1 and 2. Using a small screwdriver, rotate both screws out (counterclockwise) by one turn each. Then do the same with airflow valves number 5 and 6.

1.2.1

Too much sample lift air flow

If the sample lift airflow is excessive large, then the sample is not inserted rapidly into the BST as described in section <u>1.1.1</u> above, despite the open bypass exit hole. The samples may get stuck on top of the BST.

Reduce the sample lift airflow.

See section <u>1.1.1</u> of this manual on how to adjust the sample lift airflow for a good sample lift within 10 seconds.

If the sample lift airflow needs to be excessive large, then check section <u>1.1.2</u> and section <u>1.1.4</u> for possible faults.

Sample holder tray does not advances firmly

1.2.5

The sample holder tray must advance with some speed and should be stopped firmly in the next position. If the sample holder tray just creeps forward slowly and hesitantly, then the next openings for the sample may not be aligned properly and the sample may get stuck. Although the NMR Case is factory adjusted, this specific adjustment is air pressure dependent and therefore may need some tweaking at the installation site.

Check that the bottom of the sample holder tray is clean and free of foreign debris like sticky tape, glue from tape or staplers.

Check that the 3 white round plates located on the NMR Case base plate are clean and free of foreign debris like sticky tape, glue from tape or staplers parts.

Switch off the motion controller and push the black pin on the side to the inside out of the way. Mount the sample holder tray and check for smooth rotation by hand. It should not go easy, but with a constant force and without steps.

If the sample holder tray rotates smooth, then increase the airflow for the pneumatic piston by adjusting flow reducers # 4. It is located underneath the NMR Case base plate, on a manifold (see *Figure A.3.*). Use a small screwdriver and turn the adjustment screws counter clockwise (CCW) out by quarter turn increments. Check for the firm advancement and turn further out if necessary. If there is no change in the sample holder tray advancement, then turn flow reducer # 3 out counter clockwise (CCW) by quarter turn increments as well.

However the airflow should not be increased too much or the sample holder tray will slam the sample tubes. See section 6 of this manual.

Alignment of the adapter collar, seal ring and brown interface tube	1.2.6
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Check the alignment according section <u>1.1.6</u> of this manual.

Interface tube inside the adapter collar

Check it according section <u>1.1.8</u> of this manual.

1.2.7

Sample holder tray does not advance at all

A sample has been removed manually or the NMR Case has been used in manual mode. Make sure there is **no** sample inside the magnet. Press the RESET button while the sample lift is OFF to clear the sample tracking memory.

Increase the airflow for the pneumatic pistons by adjusting the flow reducers # 1,2,3,4. They are located underneath the NMR Case base plate, on a manifold (see *Figure A.3.*). Use a small screwdriver and turn the adjustment screws counter clockwise out by one turn each.

Open the motion controller (see section <u>1.5</u>). Observe the turn ON/OFF sequence of the green LED lights. The upper row of lights is marked IN, the lower row of lights is marked OUT.

Events	LED sequence			
Sample goes up (eject)	ON	IN - 1, 3, 2, 0		
Sample holder tray rotates	ON	OUT - 1, 0		
Sample goes down (insert)	OFF	IN - 0, 2, 3, 1		
Rotating mechanism resets	OFF	OUT - 1, 0		
Air ON (always ON)	ON	OUT - 2		
Next sample is inserted	ON	OUT - 3		
No sample is inserted	OFF	OUT - 3		
Reset button depressed	ON OFF	IN – 4 OUT - 3		

Check that IN - 0 is turned ON when the sample is on top of the sample holder tray. The sample holder (spinner) must be pressed up firmly against the sample stop head of the tray by the sample lift air. This turns on IN –0. If there is a gap between the spinner and the stop head, then IN - 0 is not turned ON and the motion controller does not rotate the sample holder tray. Increase the sample lift airflow according section <u>1.1.1</u>.

Check that IN-3 is ON before IN-0 is ON when ejecting the sample. If IN-3 is ON after IN-0, then the controller unit should be exchanged with a new version (ECL 30).

If the room temperature is above 24 degrees C and the supply main air pressure above 70 psi (5 bar), then it can happen that the green lights ON -1 and ON - 0 do come on, but the sample holder tray does not rotate. If this is the case, then the motion controller should be exchanged with a new version (ECL 30).

The sample holder tray advances when the eject air is turned OFF

1.4

The hoses for sample lift air and spinning rate air are switched around. The spinning air activates the NMR Case.

1.3

Check that the sample lift air comes out of the white hose.



Make sure there is no sample in the magnet and press the RESET button.

This clears the sample position memory. The controller will not change samples if he thinks that a sample is already down in the shim coil.

If it still does not work after pressing the RESET button, then follow the next steps.

- 1. The controller contains electrical valves, which do not work in a magnetic field. Make sure that the controller is at least 2 meters (6 feet) away from the magnet dewar cryostat.
- Open the motion controller. First loosen the four screws of the cover by two turns counter clockwise each. Now lift up the cover up as far it goes (the cover may stick to the case, just pull on it). Finally loosen the screws all the way and pull the cover off.
- 3. Switch the motion controller off and on. Observe all LED lights and check on which outputs the air comes out.

For the first two seconds the following LED's should blink and no air should come out:

		0	1	2	3	4	5		IN
Green	Green	Red	Green						
PWR	RUN	ERR	STAT	0	1	2		3	ουτ

Two seconds after switching on, air should come out of L-IN and RET, the LED's should be on (* Out 3 may be green or not):

		0	1	2	3	4	5		IN
Green	Green			Green		Green		(Green*)	
PWR	RUN	ERR	STAT	0	1	2		3	OUT

Three seconds after switching on, the air should come out of L-OUT and RET, the LED's should stay on (* Out 3 may be green or not):

		0	1	2	3	4	5		IN
Green	Green					Green		(Green*)	
PWR	RUN	ERR	STAT	0	1	2		3	Ουτ

- 4. If there are no LED lights, check the power cord and the fuse. There is a spare fuse inside the unit, 250V-3A slow-blow.
- 5. If the red LED stays on longer than two seconds, then the motion controller unit needs to be replaced.
- 6. To check the IN-LED's individually, use a short piece of Ø 4 mm hose (supplied) to blow into the corresponding sensor inputs.
- 7. To check IN-4, press the RESET button, it activates IN-4 (and switches OUT -3 off if it was on).

LED	Input	Event status	Hardware
IN-0	SENSOR 1	Sample up, on top	Pressure sensor 1.5 " H2O PSF 100 A – 1.5
IN-1	SL-SENSE	Sample lift air on ,low pressure	Pressure sensor 4" H2O PSF 200A dual 4/15
IN-2	SENSOR 2	Adapter collar pressure on	Pressure sensor 0.5 " H2O PSF 100 A – 0.5
IN-3	SL-SENSE	Sample lift air on, high pressure	Pressure sensor 15" H2O PSF 200A dual 4/15
IN-4	RESET	Reset sample insertion memory	RESET switch at the side of motion controller

- 8. Connect SENSOR 1, SENSOR 2 and SL-SENSE of the black pneumatic hose assembly to the motion controller and, at the other end, blow **simultaneously** into the three hoses no. 5, 6 and 7.
- 9. While slowly increasing the blowing pressure, observe the sequence of the IN-LED's turning green:

IN-2, IN-0, IN-1, IN-3.

10. As soon all four IN-LED's turned green, the OUT-LED's should turn on/off in the following sequence:

OUT-3 on, OUT-3 off, OUT-1 on, OUT-0 on, OUT-3 on.

11. At the same time, air should come out of the pneumatic outputs in the following sequence: RET air off, EXT air on, L-OUT air off, L-IN air on.

After releasing the blow pressure, the controller goes back to the status in section $\underline{1.5}$

LED		Output	Action	Hardware
OUT-0	On	CYL A	L-IN (Latch in)	MAC valve for CYL A (located in middle)
		Connector	Indicates tray advanced	Test connector pin 7 = + 5 V / Ground = pin 5
	Off	CYL A	L-OUT (Latch out)	
OUT-1	On	CYL B	EXT (Latch extend, rotate tray)	MAC valve for CYL B (located at left)
	Off		RET (Latch retract)	
OUT-2	On	MAIN AIR	Main air on	MAC main air valve (small, located at
	Off		Main air off	right)
OUT-3	On	Connector	Indicates sample changed.	Test connector pin 3 = + 5 V / Ground =
	Off		Can be switched off with RESET button.	pin 5

The sample holder tray slams samples when advancing

1.6

1.8

If the sample holder tray is slamming into the next position so hard that the sample tubes are shaking dangerously, then the airflow for the pneumatic piston needs to be decreased. This is done by adjusting the air flow reducer # 4, located underneath the NMR Case base plate on a manifold (see Installation manual <u>Figure A.3.</u>). Use a small screwdriver and turn the adjustment screw clockwise (CW) in by quarter turn increments. Check for slamming and turn further in if necessary.

However the airflow should not be decreased too much, else the sample holder tray will no longer advancing firmly. See section <u>1.3</u> of this manual.

The adapter collar does not fit to the shim stack	1.7
---	-----

One adapter collar for a shim upper stack type BST is included with every NMR Case.

For shim stacks other than of type BST, different adapter collars can be ordered and mounted to the NMR Case base.

Read and follow the NMR Case system requirement manual P/N B2928, <u>section</u> <u>1.6</u>.

The NMR CASE does not fit

Read and follow the NMR Case system requirement manual P/N B2928, *section* <u>1.11</u>.

The sample spin rate goes up to 150 Hz

1.9

1.10

Check the following to see if the problem goes away:

Unplug the spin rate cable connector at the BST shim stack. Check it and plug it back in.

Remove the BSMS sample control modifier cable B1428 (see instruction sheet B2300).

Check the spin rate settings.

Intermittent problems

The following 4 items need to be properly adjusted for a reliable long-term operation of the NMR Case.

Item	Necessary Adjustments	Troubleshooting Guide
1	NMR Case or magnet not leveled	Section <u>1.2.1</u>
2	Sample lift air flow	Section <u>1.1.1</u> and <u>1.1.7</u>
3	Sample holder tray movement	Section <u>1.2.5</u> or <u>1.2.6</u>
4	Seating of collar and adapter tube	Section <u>1.2.7</u>

Sample lift (eject) sometimes hangs on top of the BST and does NOT go up into the NMR Case 1.11

ltem	Check out	Troubleshooting Guide
1	Correct closing of the sample lift air bypass	Section <u>1.2.3</u>
2	Seating of collar and adapter tube	Section <u>1.2.7</u>
3	Sample lift airflow	Section <u>1.1.1</u>
4	Alignment of collar, ring, brown tube	Section <u>1.2.6</u>
5	Sample holder tray advances firmly	Section <u>1.2.5</u>
6	Sample lift airflow	Section <u>1.1.1</u>

Sample lift (eject) always hangs on top of the BST and does NOT go up into the NMR Case 1.12

Item	Check out	Troubleshooting Guide
1	Sample lift airflow	Section <u>1.1.1</u>
2	Correct closing of the sample lift air bypass	Section <u>1.2.3</u>
3	Sample holder tray advances firmly	Section <u>1.2.5</u>
4	Motion controller has no power	Section <u>1.5</u>
5	Motion controller has no air pressure	Section <u>1.5</u>

|--|

ltem	Check out	Troubleshooting Guide
1	Sample lift airflow	Section <u>1.1.1</u>
2	Sample holder tray advances firmly	Section <u>1.2.5</u>

Sample insert (down) sometimes hangs on top of the sample holder tray 1.14

Item	Check out	Troubleshooting Guide
1	Sample holder tray advances firmly	Section <u>1.2.5</u>
2	Correct opening of the sample lift air bypass	Section <u>1.2.3</u>
3	Too much sample lift air flow	Section <u>1.2.4</u>

Sample insert (down) sometimes hangs at the white ring on top of the adapter collar 1.15

ltem	Check out	Trouble Shooting Guide
1	Sample holder tray advances firmly	Section <u>1.2.5</u>

2	Alignment of collar and white ring	Section <u>1.2.6</u>
3	Seating of collar and adapter tube	Section <u>1.1.8</u>
4	BST shim with sharp edges	Section <u>1.2.2</u>
5	NMR Case or magnet not leveled	Section <u>1.1.8</u>

The sample holder tray does not advance away from position 0

1. A sample has been removed manually or the NMR Case has been used in manual mode. Make sure that there is no sample inside the magnet. Press the RESET button (on the side of the controller) while the sample lift is OFF to clear the sample tracking memory. Note: pressing the RESET button while the sample lift is ON does not work.

There was a mains power outage. See section 2.6.

Sometimes the tray rotates in the wrong direction

Refer to *Figure A.3.* at the end of this manual and locate the air flow adjustment screws number 1 and 2. Using a small screwdriver, rotate both screws out (counterclockwise) by two turns each.

Make sure that the sample holder tray rotates fast enough. Refer to section <u>1.2.5</u> of this manual and, if necessary, adjust the air flow adjustment screw number 4 out (counterclockwise) in half turn steps until the tray advances fast and comes to a firm stop (but does not slam the samples).

Check that the motion controller is not too close to the magnet, see section 1.5

If this does not solve the problem, then the motion controller should be exchanged with a new version EC 30.

Sometimes the tray does not rotate

1.18

1.16

1.17

Item	Check out	Troubleshooting Guide
1	I – 0 turn ON	Section <u>1.3</u>
2	I – 3 ON before I – 0	Section <u>1.3</u>
3	O – 1 and O – 0 ON, but no tray rotation	Section <u>1.3</u>
4	Controller too close to the magnet	Section <u>1.5</u>
5	Oil or dust in the cylinders	Section <u>2.1</u> , <u>2.2</u> , <u>2.4</u>

Sometimes samples stuck in brown interface tube (inside the adapter collar)

See *Figure A.1.* and *Figure A.2.* for location of the adapter tube.

See *figure 3* of the installation manual on how to exchange it.

Problems During Operation

2

This chapter covers problems that occur after the unit has been operating routinely for a longer time.

The air cylinder is too weak and does not rotate the sample tray completely 2.1

Actually the air cylinders are so strong that they can slam the sample tray with a large force, causing damage to the push lever, extend stop post and finally the base plate.

Therefore the air cylinders have to be restrained. This is done with airflow control valves. Each cylinder has two airflow control vales. The airflow passing through the valve can be adjusted with a screwdriver and thus the power of the cylinders can be set precisely.

So if there is not enough sample tray rotation, a quarter turn counter-clockwise (out) on the adjustment screw of the # 4 flow control valve usually solves this problem.

See *Figure A.3.*: NMR Case bottom view for the location of the airflow adjustment screws.

See installation manual B926, *Figure 6, page 24* for another view of the air flow adjustment screws.

WARNING!

Oil and dust from the compressed air source will weaken and damage the air cylinders after a while.

See the manual "NMR Case System Requirements, <u>section 1.10</u>" for the specifications of the compressed air.

Dust in the compressed air or gas line can block the small opening inside the airflow control valves. To unblock the valves, do the following:

- 1. Switch off the motion controller.
- 2. Pull off all air hoses at the cylinder ports.
- 3. Switch on the motion controller.

- 4. While the air is coming out of the open hoses, turn the screw of the airflow control valves all the way out and then back in. This clears out the small particles.
- 5. Switch off the motion controller and plug the hoses back into the ports of the cylinder.
- 6. Switch the motion controller back on and adjust the airflow control valves exactly.

See section <u>2.5</u> for other possible causes of incomplete sample tray rotation.

See section <u>2.4</u> on how to check out the air cylinders.

The air cylinder leaks oil

There should be no oil inside the cylinders. The whole NMR Case is based on oilfree air cylinders and air valves.

The oil is coming from the compressed air source. It will damage the air cylinders after a while.

All oil filters need to be flushed out and cleaned on a regular basis, otherwise they stop the filtering operation.

See the manual "NMR Case System Requirements, <u>section 1.10</u>" for the specifications of the compressed air.

To remove the oil, clean air must be blown through all hoses and valves of the NMR Case and the Motion controller. The air cylinders may need replacement.

The Bruker part number for the large Bimba cylinder A and B is 92551

The Bruker part number for the small Bimba cylinder is 92552

The NMR Case shares off sample tubes

After a mains power loss the console will set the sample lift air flow back to the stored value. If the stored value is the factory default of 750 (instead of the necessary 850), the sample will not eject all the way up to the top of the sample tray anymore, but rather stay somewhere inside the sample tray.

If this is the case, the built in safety features of the NMR Case controller prevents the advance and rotation of the sample tray and the sample is protected.

If the operator now decides (wrongly) to press the RESET button before ejecting the sample, then the safety feature of the NMR Case is overridden, the sample tray will advance and share off the sample tube.

The better way:

- Read <u>"Sample lift air flow" on page 5</u> for adjusting sample lift air flow.
- Make sure to store the larger air flow value in memory.
- The RESET button should be pressed only if a sample has to be removed manually without going into the sample holder tray.
- Only press the RESET button if there is no sample inside the magnet.

2.3

2.2

2.4

2.5

WARNING!

Oil and dust from the compressed air source will weaken and damage the air cylinders after a while. See section <u>2.1</u>.

To check the air cylinders:

- Make sure the motion controller is switched on. Remove the sample holder tray. Refer to <u>Figure A.2.</u>. Try to move manually the black push lever at the side of the base cover to the left (to position Cyl B Extend) and also try to push it in. It should require a great force to move the push lever.
- 2. Switch off the motion controller and move the push lever the same way as above. Now it should be easy to move and it should not stick at any point.

The Bruker part number for the large Bimba cylinder A and B is 92551

The Bruker part number for the small Bimba cylinder is 92552

The adapter collar cannot be removed from the BST upper stack.

The adapter collar should sit tight on the top of the BST shim stack. There is an Oring inside the collar, which causes the tight fit. There are no screws that attach the collar to the BST shim stack. Normally the collar can be pulled off by hand, using a little force.

The BST shim stack has a serial number label glued to the outside of the cylindrical top part. The glue of this label can be the reason for a very tight fit of the collar. In this case, use a piece of wood to lever off the collar from the shim stack.

WARNING!

Do not use metallic tools (screwdriver). Beware of the very strong magnetic field.

To avoid this problem in the future, remove the label carefully from the shim stack and reattach it again to the lower, narrower part of the shim stack, and apply some silicon grease to the O-ring.



The black push lever on the side of the NMR Case feels loose and does notmove at all.2.6

ltem	Check out	Troubleshooting Guide
1	Motion controller has no power	Section <u>1.5</u>
2	Fuse is blown	Section <u>1.5</u>
3	Motion controller has no air pressure	Section <u>1.5</u>







Figure A.1. Top View



Figure A.2. Side View



Figure A.3. Bottom View

Figures

1 Installation Problems	5 17
2 Problems During Operation	
A Illustrations	21
Figure A.1. Top View	21
Figure A.2. Side View	22
Figure A.3. Bottom View	23

Figures

End of Document

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