NMR Probes

Installation & Operation of 0.7/1.3/1.9 mm SB/WB Probes for Automated Insert & Eject Functionality

User Manual

1 Description

All WB & certain SB probes for small diameter rotors built from 2019 onward provide the new insert & eject functionality. This feature provides access to easy sample changes without removing the probe from the magnet system using a docking port on the bottom of the probe.

The following description is intended to describe the installation procedure (probe + MAS 3 unit) and the general operation.

Hardware prerequisites for optimum workflow integration:

- MAS 3 unit
- MAS 3 firmware version > “mas3_20191111_1111.bin”
- 1.9/1.3/0.7 mm probes equipped with the new docking port
- TopSpin version >3.6 or >4.0.5
- Compatible tubing set with additional Venturi connection (H152590)

2 Probe Installation

In general probe installations for these kinds of probes follow the same basics as any other solid-state probe.

For probes providing insert & eject the following connections must be made:

- The probe connection Venturi pressure must be connected to the Option connector of the MAS 3 unit.

This only works if the firmware version of the MAS 3 unit is “**mas3_20191111_1111.bin**” or higher, available on the FTP server, or included in TopSpin 4.0.8 or TopSpin 3.6.3. For older MAS systems the connection must be done using the “**frame cooling**” and the operation is only manual.

- The probe connection “Insert” must be connected to the corresponding port on the MAS 3 unit.
- The probe connection “Eject” must be connected to the corresponding port on the MAS 3 unit.

To be able to operate the probes a MAS 3 firmware needs to be installed which is newer than November 2019. This firmware can be found at the *ftp.bruker.ch* firmware server. With the latest TopSpin versions (>4.0.8, December 2019), the MAS 3 firmware will be upgraded automatically.

If these prerequisites are fulfilled, the probe can be operated fully automated using the MASDISPLAY user interface in TopSpin.

Customized rotation profiles may not work combined with these new Venturi probes, since the additional vacuum is not generated correctly.

PROBE	PH MASDVT850W6 BL1.9 X/Y/H +14N
KA Order No.	6290134
Ext. Order No.	424474
Part No./ Serial No.	H177715/0001
Custumer	University of Warwick
tested by / date	NFO , 06.11.2020
Delivered / date	Nov. 2020

BRUKER
NMR Probe Department

Schematic

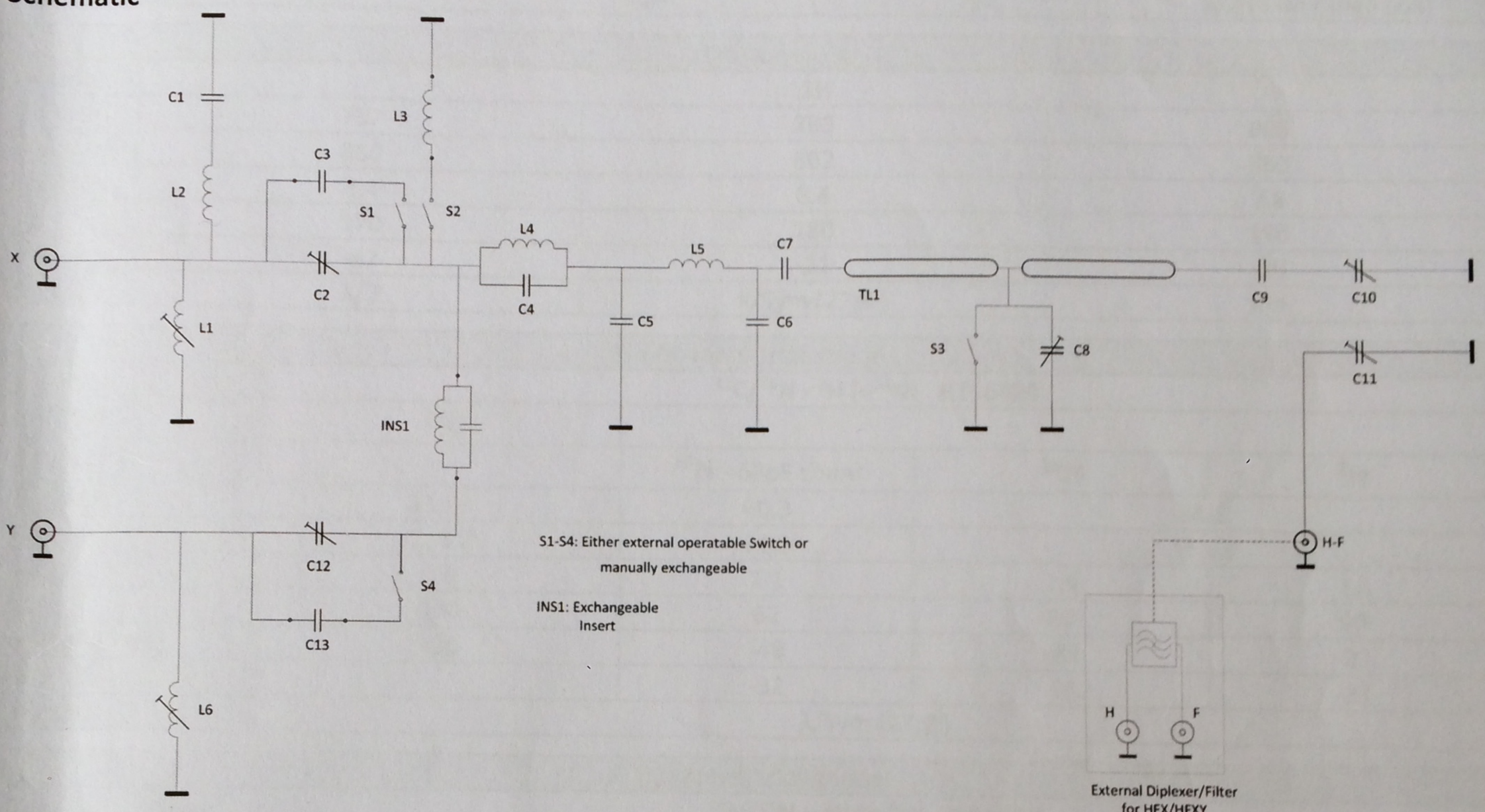

Table of Values

	Value	Part No.	Comment
C1/L2	3.3pF	86624	Capacitor + Lead
C2	1.5-52pF	1856542	X tuning
C3	———	———	
C4/L4	2.2pF / 2T	36900	1H stop circuit
C5	———	———	
C6	———	———	
C7	———	———	
C8	27pF	89172	Symmetry C
C9	5.1pF	1830022	
C10	0.8-10pF	67837	1H tuning
C11	0.8-10pF	67837	1H matching
C12	1.5-52pF	1856542	Y tuning
C13	68pF	89160	shunt for 14N
INS1			Exchangeable Insert,
L1	7T / D=7	H171666	X matching
L3	3.5T D=6		range coil
L5	8T / D=2.1	H124051	NMR COIL WB 1.9 2.12 0.5N 8T
L6	7.5T / D=7	H177906	Y Matching
TL1		H167133	transmission line
S3			λ /4 short cut screw

Measured values

X Channel Doublemode				
Nucleus	**14N**	**15N**	**13C**	**31P**
X_L / MHz	51.5		136.5	136.5
X_H / MHz	199.6		253.0	348.5
ΔF / MHz	1.7	2.5	7.7	6.3
Q	45	57	77	28
HXI / db	-36	-39	-47	-35
Mode	λ/4	λ/4	λ/2	λ/2 (with range coil)

H Channel			
Nucleus	**1H**		
X_L / MHz	757	783	806
X_H / MHz	860	862	863
ΔF /MHz	8.4	8.4	7.8
Q	170	180	190
HXI / db	-47	-31	-39
Mode	λ/2	λ/Sym (27pF)	λ/4

X/Y Channel Triplemode				
Insert	**$^{13}C/^{15}N$ - ^{2}H ($+^{14}N$) HZ16499**			
Nucleus X	**^{13}C**			
Nucleus Y		**^{14}N** +68pF shunt	**^{15}N**	**^{2}H**
F_L / MHz	213.7	60.3	68.2	
F_H / MHz	213.7	64.8	153.3	
ΔF / MHz	3.0	1.1	1.6	2.8
Q	130	62	80	90
HXI/HYI / dB	-26	-46	-33	-22
XYI /db		-32	-31	-31
Mode	λ/Sym (27pF)			

X/Y Channel Triplemode				
Insert	**$^{29}Si/^{15}N - ^{2}H$ ($+^{14}N$) HZ16569**			
Nucleus X	**^{29}Si**			
Nucleus Y		**^{14}N**	**^{15}N**	**^{2}H**
F_L / MHz	168.9	41.5		94.3
F_H / MHz	168.9	124.7		150.3
ΔF / MHz	2.0	1.2	1.8	0.8
Q	110	65	78	110
HXI/HYI / dB	-23	-27	-32	-29
XYI /db		-33	-35	-18
Mode	λ/4			λ/2

X/Y Channel Triplemode				
Insert	**$^{27}Al/^{15}N - ^{29}Si$ ($+^{14}N$) HZ16571**			
Nucleus X	**^{27}Al**			
Nucleus Y		**^{14}N** +68pF shunt	**^{15}N**	**^{29}Si**
F_L / MHz	221.5	59.3	66.9	103.4
F_H / MHz	221.5	63.8	154.3	174.8
ΔF / MHz	3.3	1.0	1.6	2.4
Q	125	62	78	105
HXI/HYI / dB	-31	-40	-66	-19
XYI /db		-23	-26	-25
Mode		λ/Sym (27pF)		λ/2

NFO, 10.11.2020

Operating Instructions for the Probe PH MASDVT850W6 BL1.9 X/Y/H (H177715/0001)

1. *Table of available X/Y combinations*

X/Y-combination	*X*	*Y*	Modification mode
	f/MHz	*f/MHz*	
^{13}C / ^{15}N - ^{2}H (HZ16499)	**213.7**	**86.1-130.5 (61.4 ^{14}N)**	λ/- C_{sym}27pF – mode, add. 68pF shunt for ^{14}N
^{29}Si/ ^{15}N - ^{2}H (HZ16569)	**168.9**	**42.0-124.0 (130.5)**	λ/ 4– mode for ^{14}N – ^{17}O , λ/ 2 – mode for ^{2}H
^{27}Al/ ^{15}N – ^{29}Si (HZ16571)	**221.5**	**86.1-168.9 (61.4 ^{14}N)**	λ/- C_{sym}27pF – mode, add. 68pF shunt for ^{14}N, λ/2 for ^{29}Si

^{13}C / ^{15}N – ^{2}H

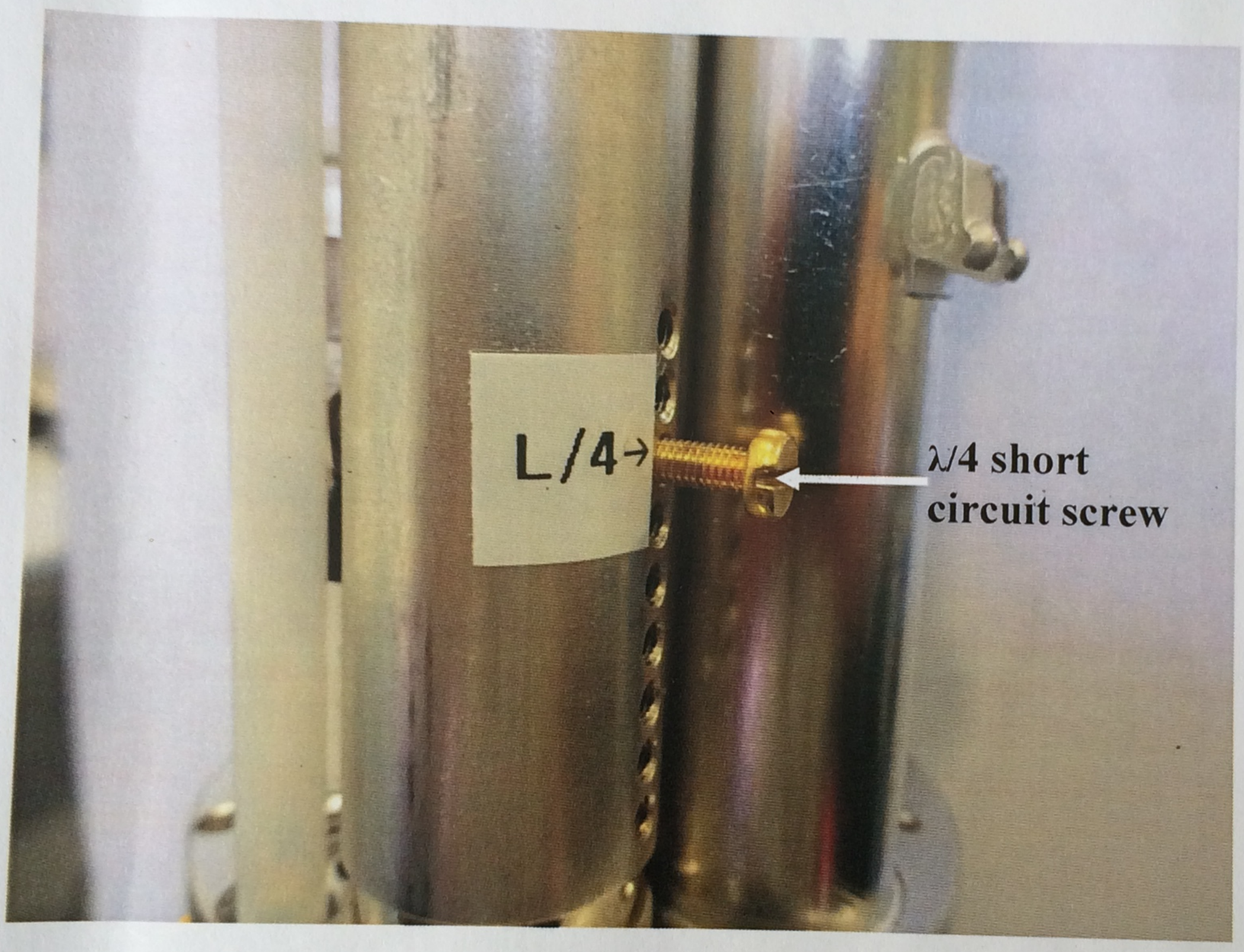

Figure 1

Screw out, but not remove, the λ/4 short circuit screw and disconnect the inner conductor of the transmission line

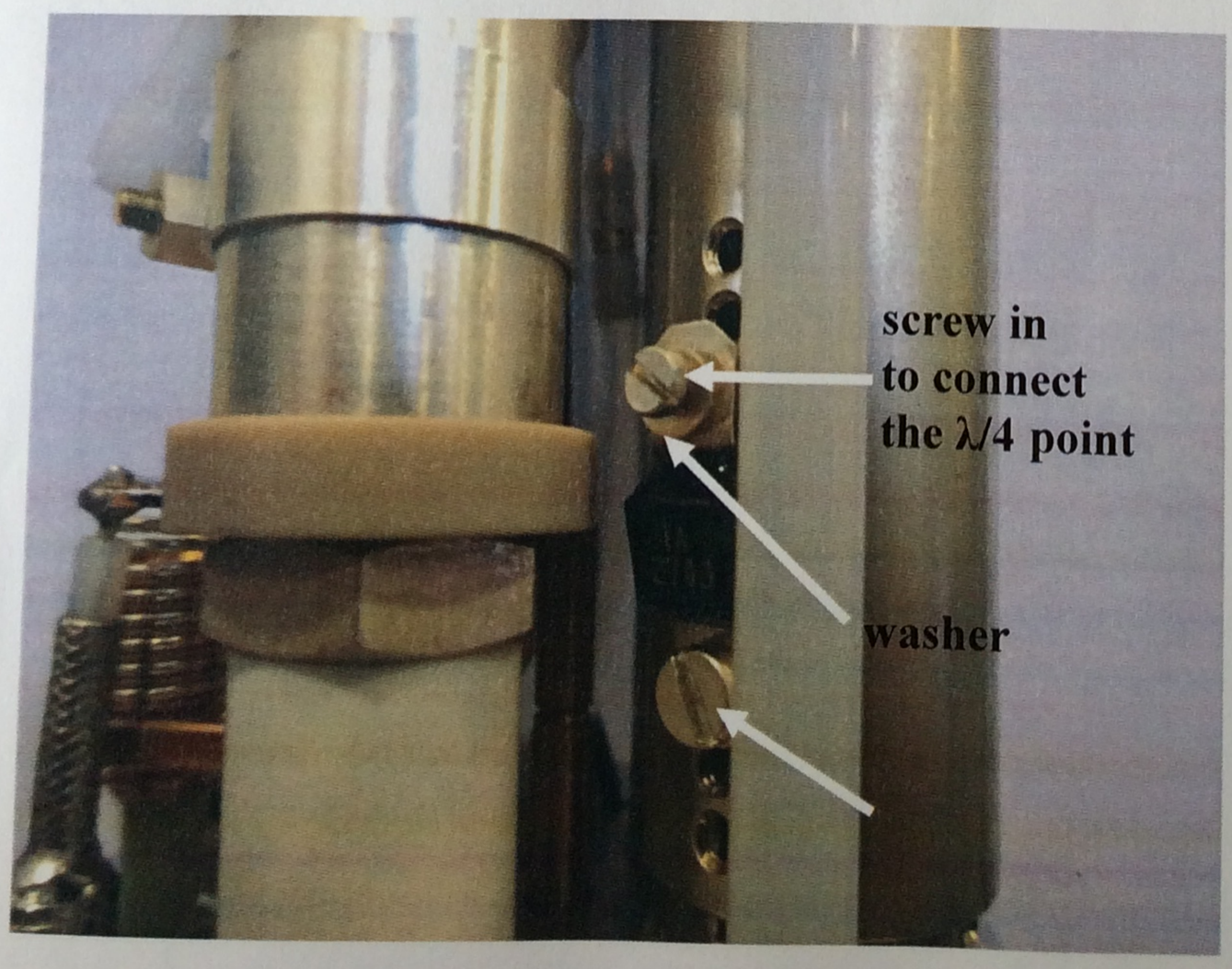

Figure 2

Screw in this screw and connect the λ/4 point with a 27pF Sym. capacitor to λ/Sym – mode.

Note:
The washer must be tight!

Do not turn this screw too far in order to avoid bending or damage of the inner conductor.

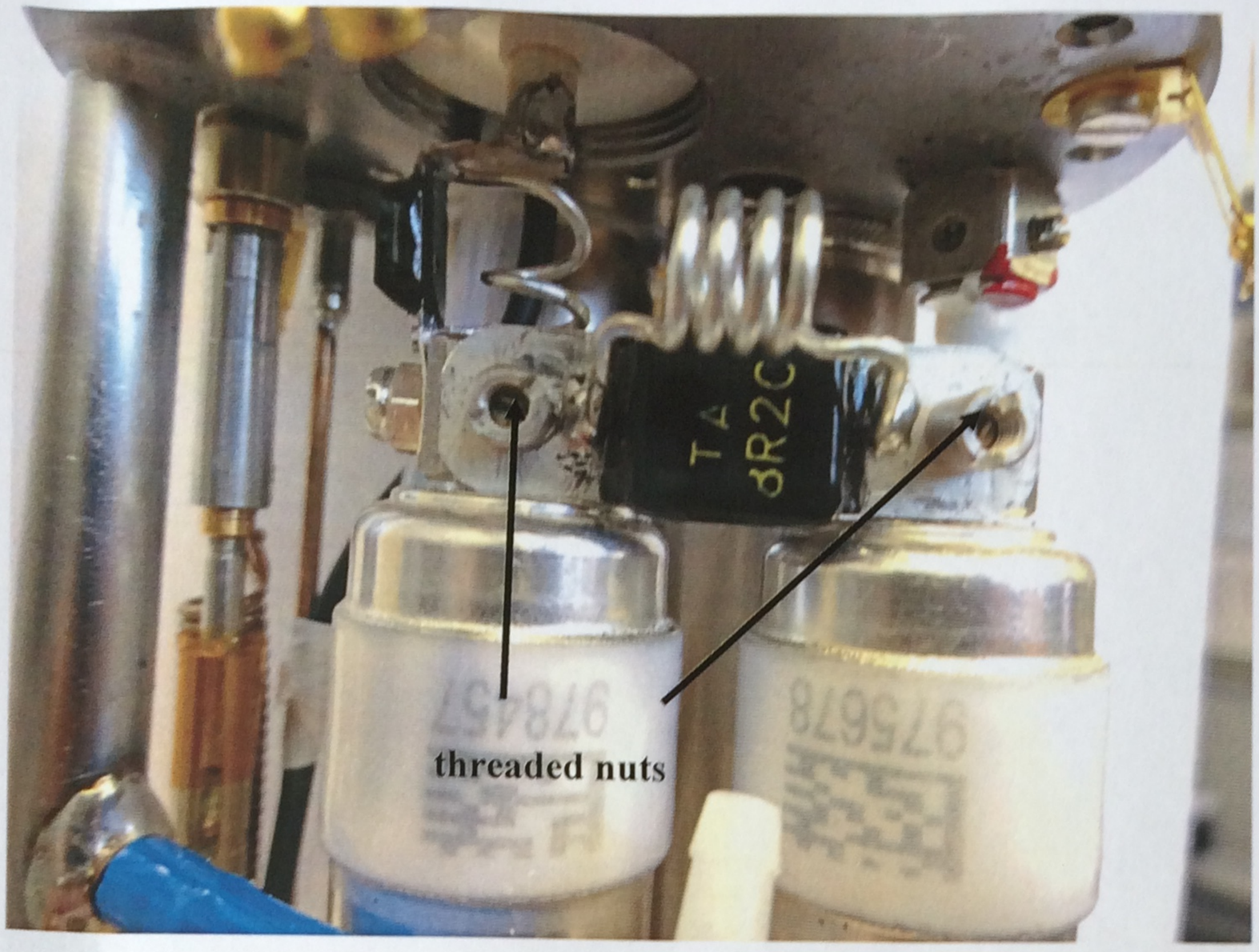

Figure 3

Install the corresponding insert as shown in the picture, in this case, ^{13}C / ^{15}N - ^{2}H and fix it with two threaded nuts

Figure 4

For ^{14}N experiments on the Y-channel install in addition a shunt capacitor of 68pF parallel to the Y tuning trimmer and fix it with two threaded nuts.

^{29}Si / ^{15}N – ^{2}H

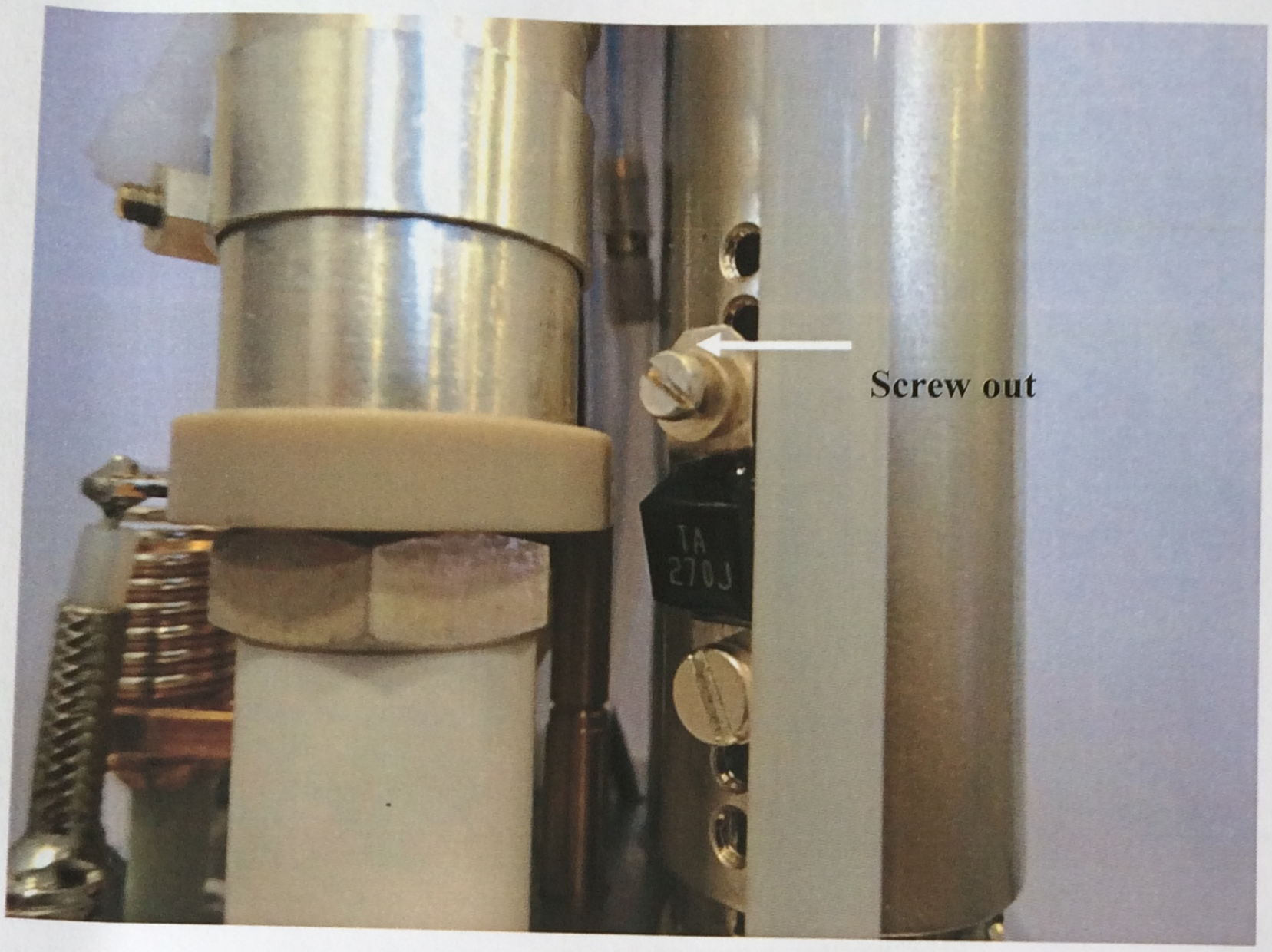

Figure 5

Screw out, but not remove and disconnect the inner conductor

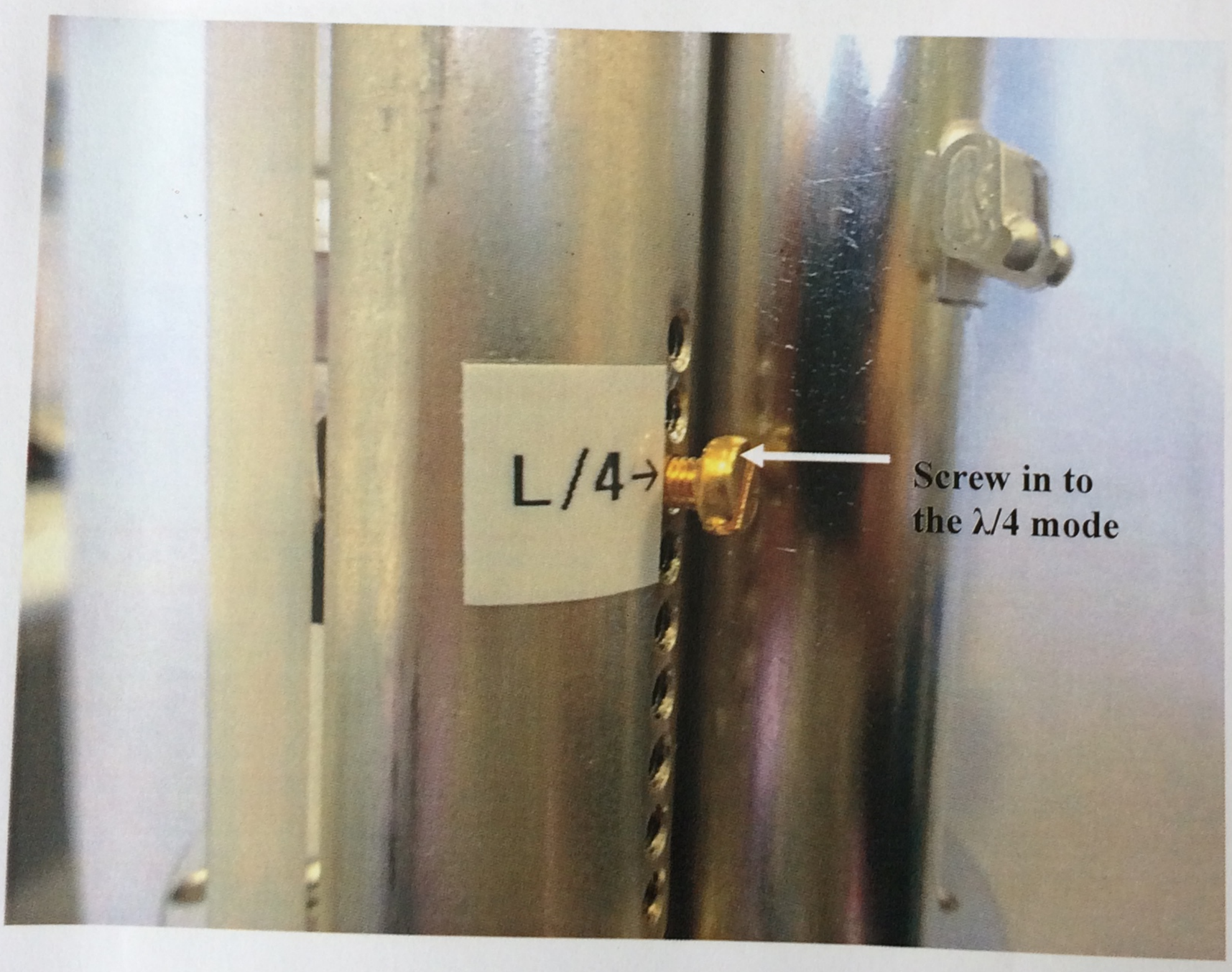

Figure 6

Screw in the short circuit screw.and connect the inner conductor of the transmission line to the λ/4 mode

Do not turn this screw too far in order to avoid bending or damage of the inner conductor.

Figure 7

Install the corresponding insert as shown in the picture, in this case, $^{29}Si/\ ^{15}N - ^{2}H$ and fix it with two threaded nuts

Note:

For experiments with ^{2}H on the Y channel change from $\lambda/4$ to $\lambda/2$ by screwing out the short circuit screw at the $\lambda/$-tube, see also figure 1

68pF shunt

Figure 8

For ^{14}N experiments on the Y-channel install in addition a shunt capacitor of 68pF parallel to the Y tuning trimmer and fix it with two threaded nuts.

^{27}Al / ^{15}N – ^{29}Si

Figure 9

Install the corresponding insert as shown in the picture, in this case, ^{27}Al/ ^{15}N – ^{29}Si and fix it with two threaded nuts

Note:

For experiments with ^{29}Si on the Y channel change from λ/sym mode to λ/2 mode by screwing out the screw at the λ/-tube, see figure 1 and figure 5

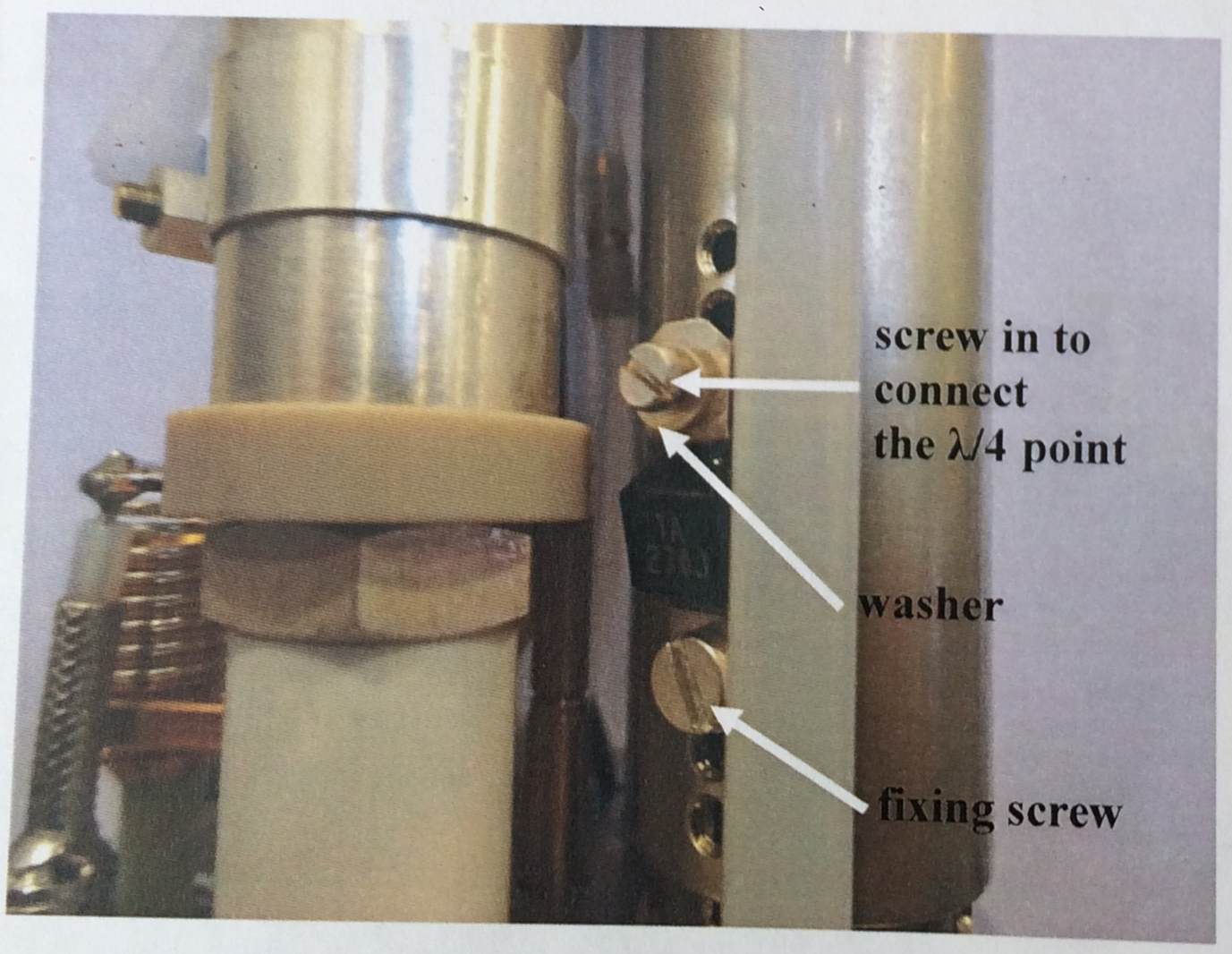

Figure 10

Screw in this screw and connect the λ/4 point with a 27pF Sym. capacitor to the λ/Sym – mode.

Note:
The washer must be tight!

Do not turn this screw too far in order to avoid bending or damage of the inner conductor.

Figure 11

For ^{14}N experiments on the Y-channel install in addition a shunt capacitor of 68pF parallel to the Y tuning trimmer and fix it with two threaded nuts.

^{31}P in Double Mode

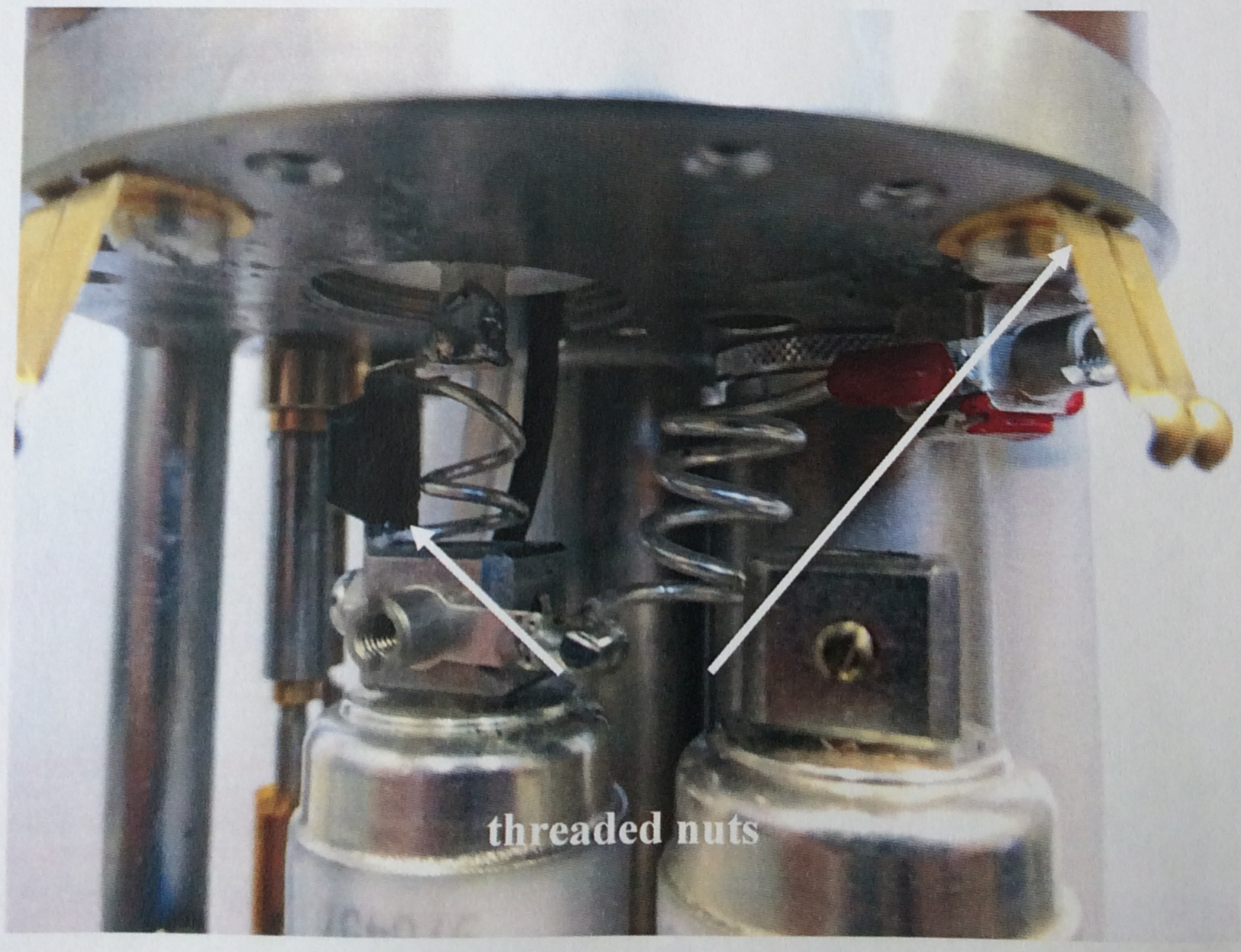

Figure 12

For ^{31}P experiments on the X channel (double mode), install a range coil in the right direction, see red marker.

Note:

Please avoid deforming the coil during installation!

3 Additional Notes

The probes can still be used if an older firmware is used, in these cases the "Venturi pressure" must be connected to the "Frame Cooling" and the pressure settings have to be controlled manually by the user in the TopSpin MASDISPLAY software (by default a pressure of 3000 mbar has to be used).

The pressure needs to be enabled BEFORE a sample insertion and must be disabled BEFORE an eject procedure.

This manual operation is also needed if the MAS unit does not support the regulated option valve as mentioned above (e.g. MAS 2).

When the latest firmware is installed, the MAS 3 unit will select the correct Venturi system and rotation profiles automatically. If the Venturi probe is changed back to a non-Venturi version the corresponding system and rotation profiles need to be selected manually on the MAS 3 Webpage.

For further information please contact: *solids@bruker.com*

4 Probe Operation

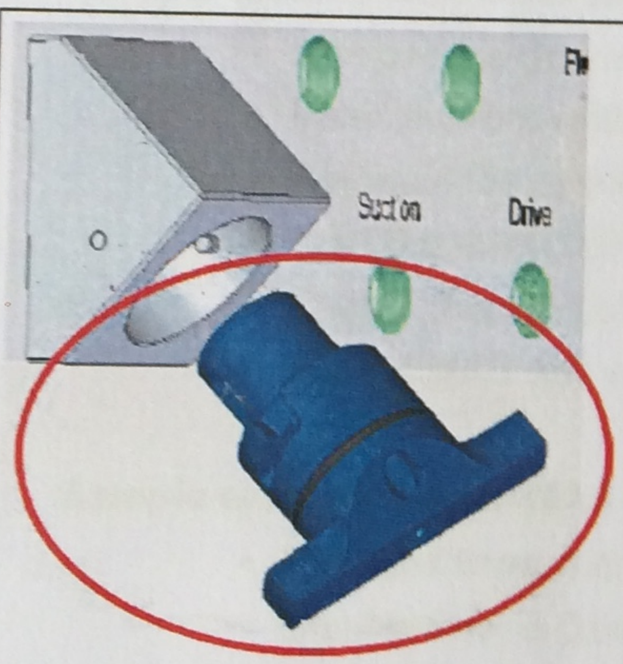

Figure 1: Docking port at probe attached to the foot case. Please make sure that the container is attached correctly with the small vent screw downwards.

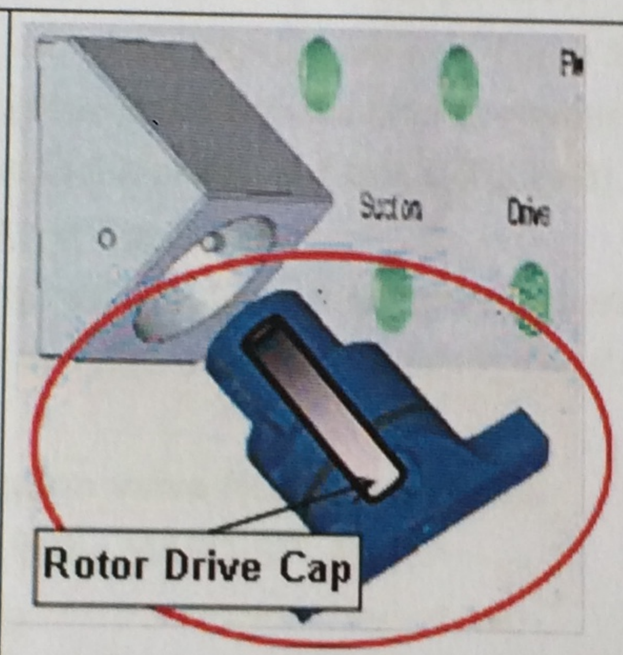

Figure 2: Rotor in docking port; rotor top cap is facing towards probe base

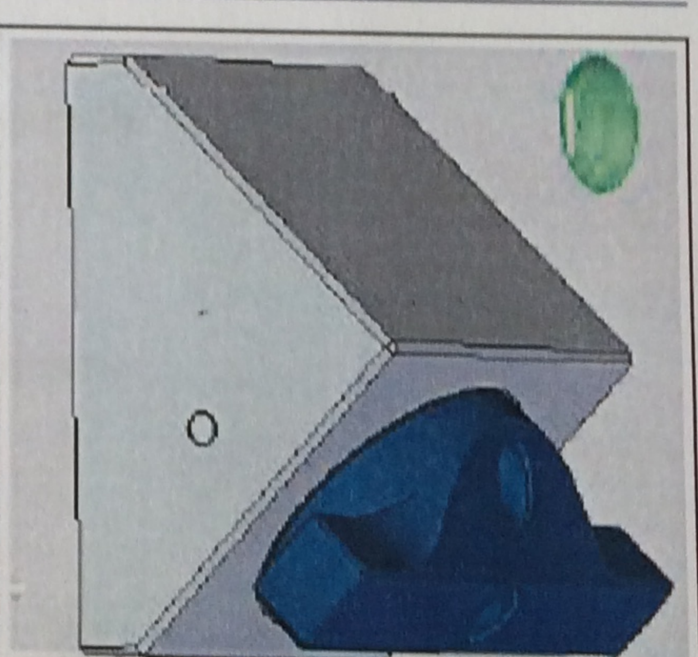

Figure 3: Remounted and locked docking port

Sample loading with recent MAS 3 support (option valve fully supported):

- Remove the docking port of the probe (blue part, figure 1).
- Insert the rotor with top cap down into the blue container (figure 2).
- Remount the container to the probe and lock it (figure 3).
- Start the MASDISPLAY in TopSpin and press **Insert**.
- Set the MAS rate according to your needs and start the rotation by pushing the **Go** button.

Sample eject with recent MAS 3 support (option valve fully supported):

- Push the **Stop** button in the MASDISPLAY.
- Push the **Eject** button and wait for the eject procedure to finish.
- Remove the docking port (figure 1).
- Remove the rotor with the corresponding tools.
- A) Remount and lock the docking port (figure 3).
  B) Insert a new rotor as described above and remount and lock the docking port.
- If a new rotor was inserted push **Insert** and **Go** buttons.

Sample loading with *older* MAS (option valve NOT supported):

- Remove the docking port of the probe (blue part, figure 1).
- Insert the rotor with top cap down into the blue container (figure 2).
- Remount the container to the probe and lock it (figure 3).
- Start the MASDISPLAY in TopSpin.
- Enable “Frame Cooling” with 3000 mbar and push **Insert**.
- Set the MAS rate and start the rotation by pushing the **Go** button.

Sample eject with *older* MAS (option valve NOT supported):

- Push the **Stop** button in the MASDISPLAY.
- Disable Frame Cooling.
- Push the **Eject** button and wait for the eject procedure to finish.
- Remove the docking port (figure 1).
- Remove the rotor with the corresponding tools.
- A) Remount and lock the rotor docking port (figure 3).
  B) Insert a new rotor as described above and remount and lock the docking port.