User's Guide

High Pressure NMR Cell

With Integrated Valve

Bruker & Agilent Cells

Version 1.06

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WARNING: This device can be dangerous and potentially harmful to users and equipment. It is very important you read and understand these instructions before using this device. A certification sheet was included with your cell indicating the maximum pressure the cell should be used. Use of the cell above this pressure could result in NMR cell failure.

There are minor differences in the shape of the Agilent versus Bruker cell. The assembly and use instructions are the same.

GUIDELINES:

DO:

- Exercise caution when pressurizing the tube. A suitable containment vessel such as a clear plastic box should be supplied to both hold the sample during preparation and for moving the NMR cell around the lab. This safety precaution is necessary to contain any fragments from a tube fracture.

- Wear proper safety equipment such as a face shield when transferring the NMR cell from the containment box to the NMR.

- Allow the pressurized sample to sit for at least 15 minutes outside the NMR to assure integrity of the cell setup.

- Check the axial alignment of the tube with the cell body, by inserting the unpressurized cell into the NMR. Once pressurized the tube cannot be adjusted to fit.

- Use a permanent marker to draw indicator marks on the tube and cell base. Maintaining the same position of the tube relative to the cell base assures consistent setup.

- Change the tube seal (TS01) after every use.

- Use care when inserting the cell into the magnet. Avoid hitting the pressurized tube against objects.

- Use the suspension chains to lower the NMR cell into the magnet in a controlled manner.

DON'T:

- Pressurize the tube above its rated limit. Remember the posted limit is the maximum pressure tested. There is no safety factor built in to that number.

- Over tighten when closing the valve. This can strip the internal components and ruin the valve.

- Over tighten the high pressure tube fitting into the valve body. This can strip the threads and ruin the valve.

- Pressurize the tube while it is in the cell setup tool. The fit of the tube is very tight. If the setup is in some way improper, the tube may shift in the setup tool during pressurization. A slight shift could fracture the tube.

FIRST USE OF THE CELL:

The NMR cell has been tested with the tube shipped. To perform this test a metal tube seat (TCSN) was placed in the cell base, and remains in place.

MATERIALS USED IN THE CELL:

The high magnetic field required for NMR demands that anything put into the magnet have no magnetic properties. Some stainless steels are considered non-magnetic, but at the high fields in use for NMR the slight magnetic properties are magnified to where stainless steel can only be used in small quantities.

These design requirements necessitate the high pressure NMR cell be fabricated from aluminum, which is relatively soft. The high pressure fittings used typically with this cell are made from stainless steel. Over tightening the fitting in the valve can strip the threads and ruin the valve. **Only tighten 1/8 turn past snug for proper sealing conditions.**

The needle in the valve is made from 316L stainless steel. This does have some magnetic characteristics, but does not inhibit its use in high field magnets in such small quantities. The strength of stainless steel is necessary due to the high forces that are applied to this piece. The stem used to move the needles is made from an aluminum alloy 642. This is slightly stronger than plain aluminum, but contains copper which has been found to leach into samples under certain conditions so cannot be used in wetted parts. The stem material is softer than 316L stainless steel so over tightening the valve when closing it can strip the stem. **Only turn the valve 1/8 turn past snug to close.** This will extend the life of the valve.

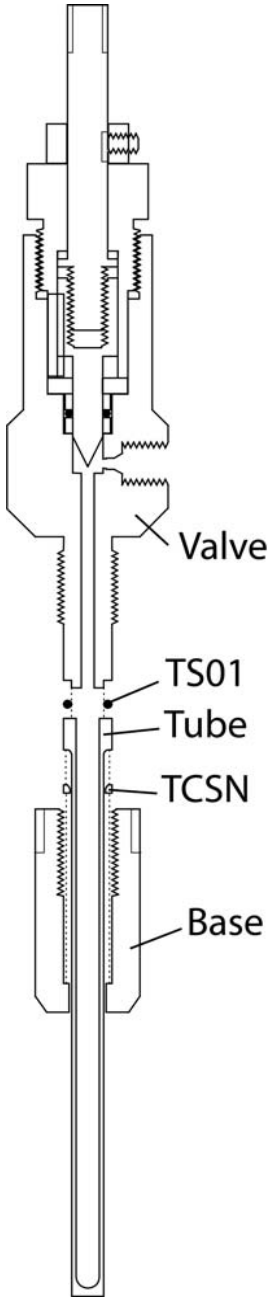
The dynamic seal used in the valve is made from Teflon. It may eventually wear out and need to be replaced. The section titled *Valve Maintenance* covers how to replace this seal. One is included with the valve.

CELL SETUP:

The cell was designed so that the tube can be easily removed and cleaned if necessary. As such the seal (TS01) is single use. An ancillary component called the tube seat (TCSN) serves as a cushion between the ceramic tube and metal surface of the cell. The drawing at the right shows the relative position of the tube seal (TS01) and tube seat (TCSN).

To assemble the cell the tube seat is placed around the tube along the bottom of the head portion of the tube. The internal beveled edge of the tube seat should be towards the head portion of the tube. This assembly is then placed into the cell base piece. The TCSN component is a permanent piece. This step should not be necessary unless the TCSN component should dislodge from the base. Otherwise it should remain in position at all times.

The primary seal is provided by the part TS01. **The seal is single use only.** For setup it should be placed on the end of the valve section piece. This assembly should then be threaded into the base. Using the 7/8” wrench for the valve, and the 1/2” wrench (Bruker) or 5/8” wrench (Agilent) for the base, tighten to set the seal. **The recommended torque required for setting the seal is 90 in.-lbs. (11.3 Nm).**



Do not exceed the maximum torque setting. This could damage the NMR cell or tube. See Cell Torque Settings section for more information.

Application of light-grade machine oil to the threads will reduce friction and provide a more accurate torque reading.

For optimum positioning of the tube the Cell Setup Tool should be used. This tool helps keep the tube axially aligned with the NMR cell. Improper positioning of the tube can prevent the cell from inserting into the NMR. To use the setup tool, first place the base with the tube already inserted. The fit is snug, so the tube may need to be pushed into position. The valve with the seal is then threaded into the base, and tightened just to the point of resistance.

Optimum tube alignment is achieved by iterative cycles of slight tightening followed by several full rotations of the cell setup tool while holding the cell static. This minimizes any tube misalignment attributable to the tool itself. Tightening during the first several cycles should be by hand followed by several more cycles using the wrenches. Finally the wrenches can be used to complete the tightening process.

A pressurized tube cannot be readjusted without releasing the pressure first and potentially losing the sample so be certain to check the alignment before continuing.

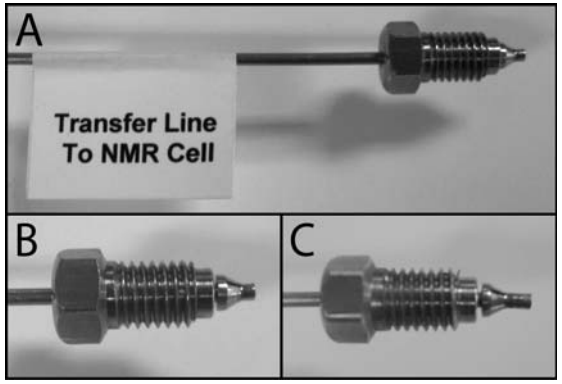
Once the setup of the cell has been completed it should be checked for proper fit in the NMR before pressurizing the sample.

After confirming the proper axial alignment of the tube with the cell, the included high pressure transfer line can be attached and sample preparation can take place.

TRANSFERING SAMPLE INTO THE CELL:

An assembled transfer line has been included with the cell. This has been fabricated exclusively for use with the NMR cell. The length of this line is 12” contributing an additional 139 µl to the system volume. See Specifications section for NMR cell volume. The same transfer line should be used, in the same orientation, with the same cell. This will provide optimal sealing, and decrease the likelihood of damage to the sealing surfaces.

The transfer line has a specific orientation that should be maintained for every use. The transfer line as shipped, shown in the figure below, panel A, has a label fixed to the end that connects to the NMR cell. For optimal performance the label should be oriented in the same direction each use; e.g. pointing down. The fitting assembly on this end differs from the other in that the 1/16” tubing protrudes less from the compression gland (compare panels B to C). This decreased length is required to keep the tubing from hitting the internal needle and possibly preventing the valve from working.



Using the transfer line in the wrong orientation will damage the NMR cell, and possibly prevent proper sealing.

INSERTING THE CELL INTO THE NMR:

Once the sample has been prepared the valve needs to be closed. Attach the handle to the stem, tightening the set screw with the 3/16” hex driver. One

consideration when closing the valve is the displacement of the needle. Depending how far the valve needle has been retracted, the volume displaced by closing the valve can be quite large relative to the volume of the tube. **It is best if the cell remains open to the pressure source while closing the valve.** This will keep the internal pressure equal to the target pressure, and also prevent potential over pressurization that could occur. **Remove the valve handle after closing the valve.**

When closing the valve, the needle displaces volume. Keeping the cell connected to a regulated external pressure source will allow the system to regulate while closing the valve. This yield a more accurate reading of the pressure inside the NMR tube.

Attach one end of the suspension chain to each clip at the top of the valve, as shown in the picture below. The NMR cell is now ready to be carefully lowered into the magnet using the chains.

Be sure to keep the pressurized cell inside the protective box when moving the cell. This keeps the user safe as well as minimizes the chance of hitting the tube against objects.

CELL TORQUE SETTINGS:

To properly set the seal (TS01) the seal must be compressed a specific amount. To do this the cell must be assembled using sufficient torque. The recommended numbers are show below:

90 in.-lbs. (11.3 Nm)	*typical*
105 in.-lbs. (14.7 Nm)	*maximum*

Using more torque does not assure a better seal and could cause damage to the cell or fracture of the tube. If the cell is leaking disassemble and try the following before applying additional torque:

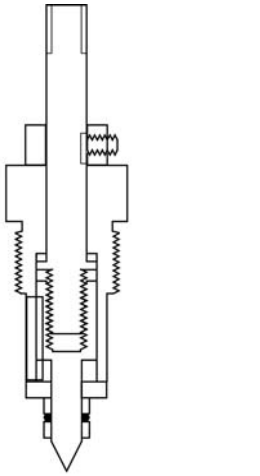
1) Make sure the sealing surfaces on the cell and tube is dry. Replace the seal.
2) Use a small quantity of light-weight oil on the cell threads to reduce friction and assist in assembly.

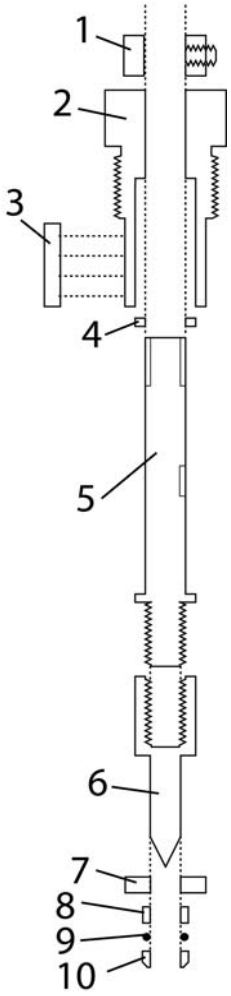
VALVE MAINTENANCE:

The needle of the valve is sealed in place with a Teflon packing o-ring. This is a dynamic seal such that the needle slides past the ring during valve actuation. Eventually, this motion may wear out the o-ring. Unfortunately, the wear and tear can be highly variable such that the evidence that this needs to be done will be the high pressure gas leaking out the top of the valve past the stem. The procedure to replace the valve o-ring makes reference to the *Valve Assembly Diagram* figure later in this booklet. Proceed as follows:

1) Open the valve as far as it will go, to fully withdraw the needle. If the valve assembly is removed with the valve closed it will damage the sealing surface and possibly decrease performance.
2) Use the 7/64” hex wrench to remove the chain clips on the stem nut.
3) Using the 5/8” wrench on the stem nut and 7/8” wrench on the valve body to loosen the stem nut, and extract the valve assembly. Be careful not to lose the needle key.
4) Remove the part labeled as the spacer in the diagram, and clean any residual o-ring material that may have built up on the inside surface of the part. The top spacer may come out in Step (3). If the spacer slides easily along the needle it can remain in place.
5) Remove the old Teflon o-ring with a hook or forceps, taking care not to score the walls of the valve. The bottom beveled spacer does not need to be removed.
6) Slide the new Teflon o-ring over the needle, and reassemble the valve assembly with a new o-ring as shown in the diagram. Be sure the needle is fully withdrawn into the stem nut. Again, if the needle is extended too far when the stem nut is tightened it could damage the sealing surface inside the valve.
7) Again, using the 5/8” wrench on the stem nut and the 7/8” wrench on the valve body, tighten the stem nut.
8) Reattach the chain clips with the 7/64” hex wrench.

VALVE ASSEMBLY DIAGRAM:





Valve Assembly
1. Stem collar
2. Stem nut
3. Needle key
4. Stem washer
5. Stem
6. Needle
7. Pressure disc
8. Spacer
9. Needle seal
10. Beveled spacer

SPECIFICATIONS:

Wetted parts valve open	7068 Aluminum, 316 stainless steel, 642 bronze, zirconia (NMR tube), Teflon, Viton
Wetted parts valve closed	7068 Aluminum, 316 stainless steel, zirconia (NMR tube), Viton
Internal volume with valve closed (3.6 mm ID tube)	**Bruker:** 1.011 ml **Agilent:** 1.067 ml
Valve needle displacement	16 µl / full turn
Pressure range	1 kbar maximum (max determined by tube)
Manifold to tube seal	Viton seal standard; single-use
Pressure connection	Manifold port is HiP AF1 (1/4”-28 UNF) for use with 1/16” tubing.
Allowed fluids	All fluids compatible with the wetted parts can be used in the cell. Examples are water, alcohols, alkanes, carbon dioxide, and xenon. Some solvents may cause buildup of aluminum oxide on the NMR manifold.

FURTHER INFORMATION:

This document may be updated periodically to reflect questions from users. Please check back at www.daedalusinnovations.com in the support section for more recent versions of this document.

Technical support can also be obtained by emailing questions to support@daedalusinnovations.com, or contacting Daedalus directly at 610-358-4728.

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