User's Guide

High Pressure NMR Cell

With Integrated Valve

Bruker & Varian Cells

Version 1.02

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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 Varian versus Bruker cell. The assembly and use instructions are the same.

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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.

- Change the tube seat (TCSN) after every ten uses.

- 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 tube seat (TCSN) was placed in the cell base, and remains in place. It does not need to be replaced prior to use.

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 Viton rubber. It will eventually wear out and need to be replaced. The section titled *Valve Maintenance* covers how to replace this seal.

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. This component deforms over time and will need to be

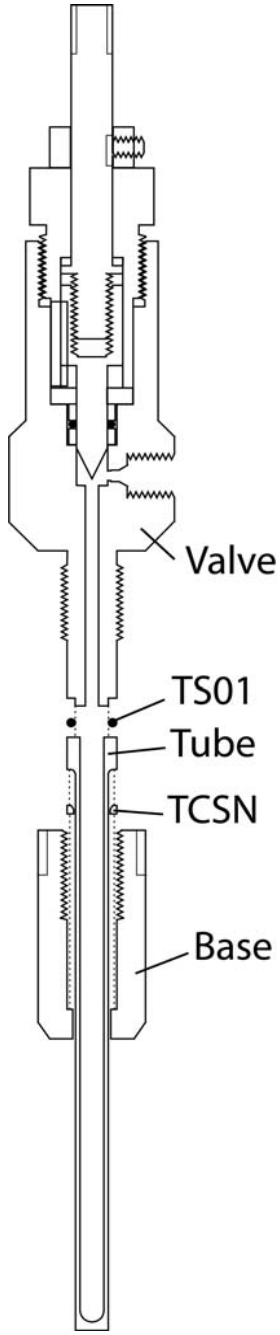
replaced after about ten uses (see *Valve Maintenance)*. The drawing at the right shows the relative position of the tube seal (TS01) and tube seat (TCSN).

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. If the tube seat does not fit it may be necessary to sand the edges with fine sandpaper.

This positioning of the tube seat is only necessary the first time a tube seat is used. **Once set, the tube seat should not be removed between uses.**

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 (Varian) for the base, tighten to set the seal.

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.

Once the setup of the cell has been completed it should be checked for proper fit in the NMR before pressurizing the sample. **Remember, a pressurized tube cannot be readjusted without releasing the pressure first and potentially losing the sample.**

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

INSERTING THE CELL INTO THE NMR:

Once the sample has been prepared the valve needs to be closed. Attach the knob to the stem, tightening the set screw with a screw 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 knob after closing the valve.**

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.

VALVE MAINTENANCE:

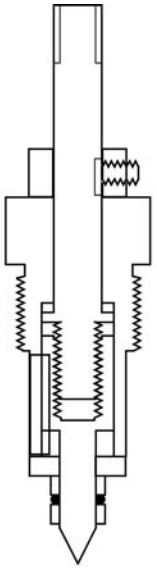
The tube seat (TCSN) should be replaced after about 10 uses. This is to prevent compounded distortion of this piece from altering the integrity of the setup. To remove the tube seat, use a small blade screw driver or small hook tool to pry it loose and remove. Replace the tube seat as described in the *Cell Setup* section.

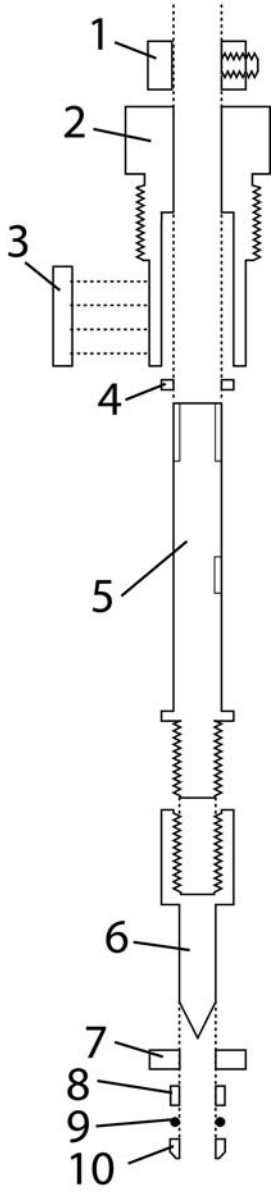
The needle of the valve is sealed in place with a Viton o-ring. This is a dynamic seal such that the needle slides past the ring during valve actuation. Eventually, this motion may wear out the o-ring. Unfortunately, the wear and tear can be highly variable such that the evidence that this needs to be done will be the high pressure gas leaking out the top of the valve past the stem. The o-ring can be replaced when a more thorough cleaning of the valve is done, which may be necessary depending on the reagents used in the samples. The procedure to replace the valve o-ring makes reference to the *Valve Assembly Diagram* figure later in this booklet. Proceeds as follows:

1) Open the valve as far as it will go, to fully withdraw the needle. Upon actuation the needle only moves up and down; it does not rotate. This means the position of the stem nut relative to the valve body should be constant to maintain an identical sealing surface. It also means that if this procedure is performed with the valve closed it could damage the needle sealing surface, and degrade the performance of the valve.
2) Use a marker to indicate a reference position on the stem nut relative to the valve body. This will serve as a guide when retightening the stem nut.

3) Use the 7/64” hex wrench to remove the chain clips on the stem nut.
4) 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.
5) Remove the beveled spacer and the old o-ring from the assembly. The beveled spacer and o-ring will sometimes not come out with the assembly. In this case remove the o-ring with a hook or forceps, taking care not to score the walls of the valve. Tap the valve on a cushioned hard surface to remove the beveled spacer.
6) 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. Depending on the degradation of the o-ring, this part may be very difficult to remove. If this is the case withdraw the needle as far as it will go, and pry loose the spacer with a flat-head screw driver.
7) Make sure the inside surfaces of both spacers are free of seal material, and reassemble the valve assembly with a new o-ring as shown in the diagram.
8) 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.
9) Hold the assembly with the stem pointing down, reinsert into the valve body, and screw into place.
10) Again, using the 5/8” wrench on the stem nut and the 7/8” wrench on the valve body, tighten the stem nut until the reference lines on the stem nut and valve body from Step (2) line up.
11) 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

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 267-499-2013.

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