

User's Guide
High Pressure NMR Cell
With High Pressure Tether
Bruker & Varian Cells

Version 1.03

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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.
- Pretest the tube at the target pressure 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.
- Leave the high pressure tubing tether attached to the cell between uses. Constant reseating of the fitting will reduce the seal integrity.
- 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 a torque of 190 in.-lbs. (21.47 Nm) when assembling the cell to assure proper setting of the seal.
- Use care when inserting the cell into the magnet. Avoid hitting the pressurized tube against objects.

DON'T:

- Pressurize the tube above its rated limit. Remember the posted limit is the maximum pressure the cell should be used.
- Insert a fully pressurized tube into the magnet. It is preferable, especially when working near the posted maximum, to insert the tube first then take it to pressure. It is recommended the tube pressure be below 5,000 psi when inserting or removing the cell from the magnet.
- Over tighten the high pressure tube fitting into the cell body. This can strip the threads or damage the seal surface and ruin the cell.
- 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.
- Use metal needles to fill the tube. The tube is made from a hard ceramic that can scrape metal off into the sample.

FIRST USE OF THE CELL:

The NMR cell has been tested with the tube shipped. A permanent tube seat (TCSN-M) was placed in the cell base prior to shipping, and should not be removed.

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 bronze or 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 cell can strip the threads or deform the seal surface and ruin the cell. It is recommended that once the high pressure tether line is attached it remain so. Constant reseating of the fitting will eventually degrade sealing performance.

CELL SETUP:

The high pressure tether line should be attached prior to full assembly of the cell. The tubing used for this purpose is 1/8" O.D., with a standard high pressure fitting with 1/2"-20 threads. A collar is

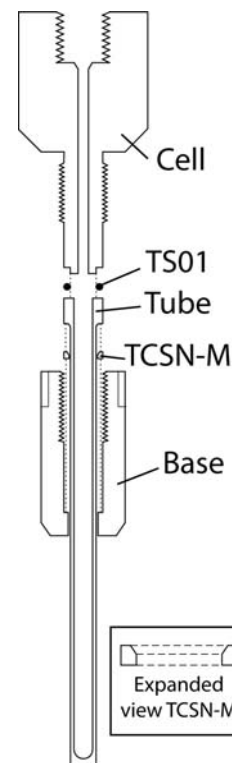


threaded onto the tubing as shown in the picture. The coned end of the tubing mates with the tapered hole in the cell. Therefore, be sure the collar is threaded far enough that these two surfaces will meet otherwise the seal integrity could be poor. Once attached this tether should not be removed between uses since doing so will likely lead to degradation in the performance.



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-M) 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-M).

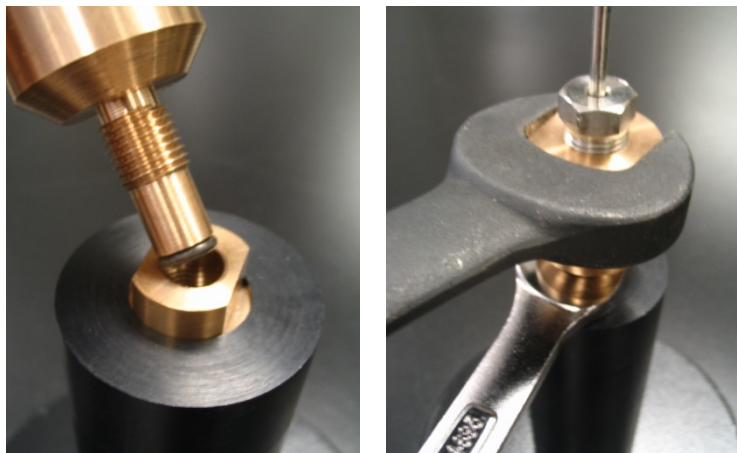
The tube seat is a permanent component, but if it dislodges from the base it should be reinserted according to the diagram. The tube seat has an internal beveled edge or chamfer. This surface should be positioned towards the head portion of the tube and this assembly then placed into the cell base piece to reposition the seat.



Once in place, 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 proper 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 sample should be added at this point using a glass pipette.** The fit is snug, so the tube may need to be pushed into position. The cell 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. During the first several cycles tighten by small increments, followed by several more cycles using larger increments. Finally, tighten to the proper torque setting.

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.**

INSERTING THE CELL INTO THE NMR:

The high pressure tether line is made of 316 stainless steel tubing. This rigid tether can sometimes make inserting the sample into the magnet a difficult process. For this reason it is strongly advised not to insert a tube pressurized over 5,000 psi. The tubes are themselves robust, but striking the tube against the magnet while pressurized near its maximum could potentially cause a tube failure.

To facilitate insertion, the high pressure tether should be straightened for a length equivalent to the depth of the magnet bore. Be sure to keep the pressurized cell inside a protective box when moving the cell. This keeps the user safe as well as minimizes the chance of hitting the tube against objects.



The cell can now be pressurized to the desired working pressure.

TECHNICAL DETAILS:

Torque Settings for cell assembly:

190 in.-lbs. (21.47 Nm)	<i>typical</i>
210 in.-lbs. (23.73 Nm)	<i>maximum</i>

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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