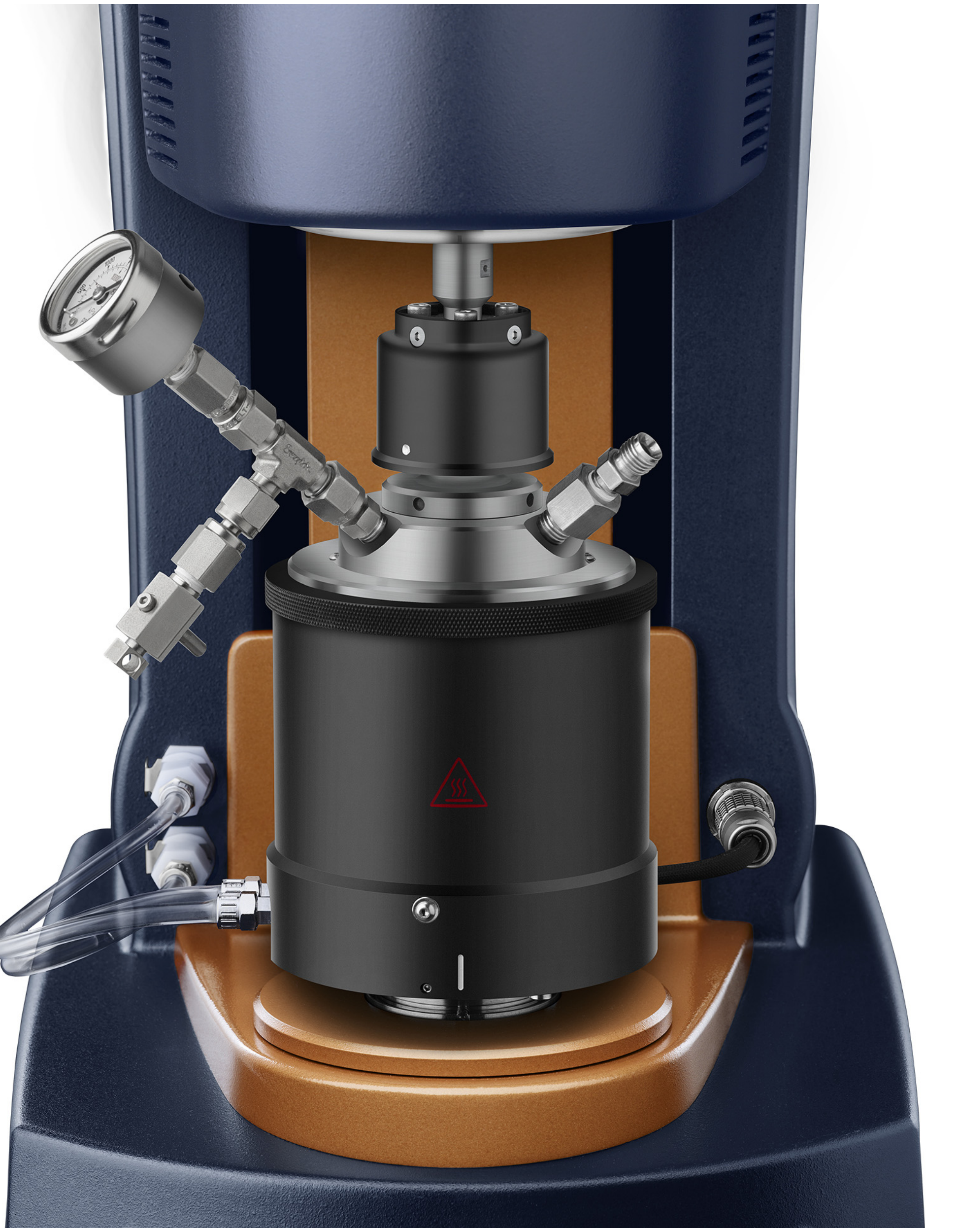
DHR Series and AR Series

Pressure Cell



Getting Started Guide

Revision E Issued May 2015

Notice

Introduction

Important: TA Instruments Manual Supplement

Please click the TA Manual Supplement link to access the following important information supplemental to this Getting Started Guide:

- TA Instruments Trademarks
- TA Instruments Patents
- Other Trademarks
- TA Instruments End-User License Agreement
- TA Instruments Offices

Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions. In the body of the manual these may be found in the shaded box on the outside of the page.

NOTE: A NOTE highlights important information about equipment or procedures.

CAUTION: A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.

MISE EN GARDE: UNE MISE EN GARDE met l'accent sur une procédure susceptible d'endommager l'équipement ou de causer la perte des données si elle n'est pas correctement suivie.

A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

Un AVERTISSEMENT indique une procédure qui peut être dangereuse pour l'opérateur ou l'environnement si elle n'est pas correctement suivie.

Regulatory Compliance

Safety Standards

For Canada

CAN/CSA-C22.2 No. 61010-1 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements.

CAN/CSA-C22.2 No. 61010-2-010 Particular requirements for laboratory equipment for the heating of materials.

For European Economic Area

(In accordance with Council Directive 2006/95/EC of 12 December 2006 on the harmonization of the laws of Member States relating to electrical equipment designed for use within certain voltage limits.)

EN 61010-1:2001 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

EN 61010-2-010:2003 Particular requirements for laboratory equipment for the heating of materials + Amendments.

For United States

UL61010-1:2004 Electrical Equipment for Laboratory Use; Part 1: General Requirements.

UL61010A-2-010:2002 Particular requirements for laboratory equipment for the heating of materials + Amendments.

Electromagnetic Compatibility Standards

For Australia and New Zealand

AS/NZS CISPR11:2004 Limits and methods of measurement of electronic disturbance characteristics of industrial, scientific and medical (ISM) radio frequency equipment.

For Canada

ICES-001 Issue 4 June 2006 Interference-Causing Equipment Standard: Industrial, Scientific, and Medical Radio Frequency Generators.

For the European Economic Area

(In accordance with Council Directive 2004/108/EC of 15 December 2004 on the approximation of the laws of the Member States relating to electromagnetic compatibility.)

EN61326-1:2006 Electrical equipment for measurement, control, and laboratory use-EMC requirements-Part 1: General Requirements. Emissions: Meets Class A requirements per CISPR 11. Immunity: Per Table 1 - Basic immunity test requirements.

For the United States

CFR Title 47 Telecommunication Chapter I Federal Communications Commission, Part 15 Radio frequency devices (FCC regulation pertaining to radio frequency emissions).

Safety

Do not attempt to service this cell, as it contains no user-serviceable components.

Required Equipment

While operating this accessory, you must wear eye protection that either meets or exceeds ANSI Z87.1 standards. Additionally, wear protective clothing that has been approved for protection against the materials under test and the test temperatures.

Instrument Symbols

The following label is displayed on the accessory for your protection:

Symbol	**Explanation**
	This symbol indicates that a hot surface may be present. Take care not to touch this area or allow any material that may melt or burn come in contact with this hot surface. Ce symbole indique la présence possible d'une surface chaude. Prenez soin de ne pas toucher cette zone ou de laisser un matériau susceptible de fondre ou de brûler entrer en contact avec cette surface chaude.

Please heed the warning labels and take the necessary precautions when dealing with these areas. This *Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

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Chapter 1:

Introducing the Pressure Cell

Overview

WARNING: TA Instruments' Pressure Cell is designed for use at temperatures up to 150°C and pressures up to 138 bar (2000 psi). At all times during the use of the cell, wear safety glasses and clothing that afford adequate protection against the sample under test and the temperature/pressure used. At other than ambient temperature, the outer surfaces of the cell may become very hot or cold. When operating at these temperatures, wear gloves that afford adequate protection against the surface temperature of the Pressure Cell and its fittings.

AVERTISSEMENT: La cellule à pression de TA Instruments a été conçue pour être utilisée à des températures allant jusqu'à 150°C maximum et des pressions de 138 bar (2000 psi) maximum. Pendant l'utilisation de la cellule, portez toujours des lunettes et des vêtements de sécurité qui assurent une protection adéquate contre l'échantillon en cours d'essai, la température et la pression utilisées. Sous d'autres températures ambiantes, les surfaces externes de la cellule peuvent devenir très chaudes ou froides. Lorsque vous travaillez sous ces températures, portez des gants qui assurent une protection adéquate contre la température de surface de la cellule à pression et ses raccords.

The Pressure Cell is used with the standard concentric cylinder, Peltier-controlled heating jacket on the DHR Series and AR-G2/AR2000ex. A copper sheath is fitted to the cell to ensure good heat transmission between the jacket and the cell.

The Pressure Cell may be used either in self-pressuring mode, in which the pressure is produced by the volatility of the sample, or in external pressurization mode, with an applied pressure of up to 138 bar (2000 psi).

NOTE: For external pressurization, the user of the cell is required to provide a high-pressure source, and suitable pressure-rated connections to a 1/8-inch or 1/4-inch NPT female fitting.

Pressure Cell Components

The Pressure Cell consists of four main component assemblies. These components include the Pressure Cell cup, the concentric cylinder rotor, the magnet assembly, and the pressure manifold. Figure 1 shows a schematic cross section of the Pressure Cell cup, rotor, and magnet assemblies, and Figure 2 shows a fully configured Pressure Cell installed on an AR Rheometer. The following section discuss these four components individually.

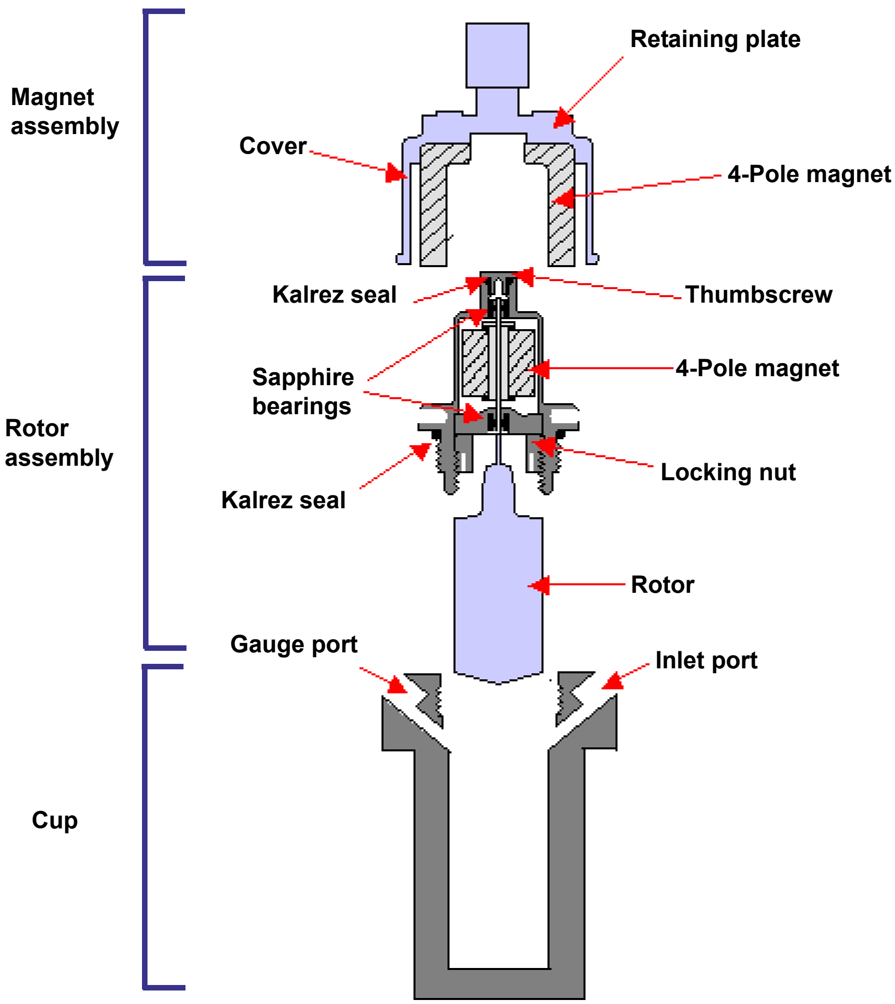


Figure 1 Cross-section schematic of Pressure Cell.

Figure 2 Pressure Cell fully installed.

The Pressure Cell Cup

The Pressure Cell cup contains the sample fluid. It is inserted into the Peltier jacket, which mounts on the rheometer using the Smart Swap™ connection. A copper sheath ensures good heat transmission between the jacket and the cup. There are three ports on the cup, which are identified by engraved labels.

NOTE: When installing NPT fittings use Teflon® thread sealing tape.

CAUTION: Do not attempt to attach or detach any fittings to or from the cell while it is mounted on the rheometer. Doing so can cause damage to the instrument.

MISE EN GARDE: N'essayez pas de fixer les raccords à la cellule ou de les en séparer lorsqu'elle est montée sur le rhéomètre. Cela peut causer des dégâts à l'instrument.

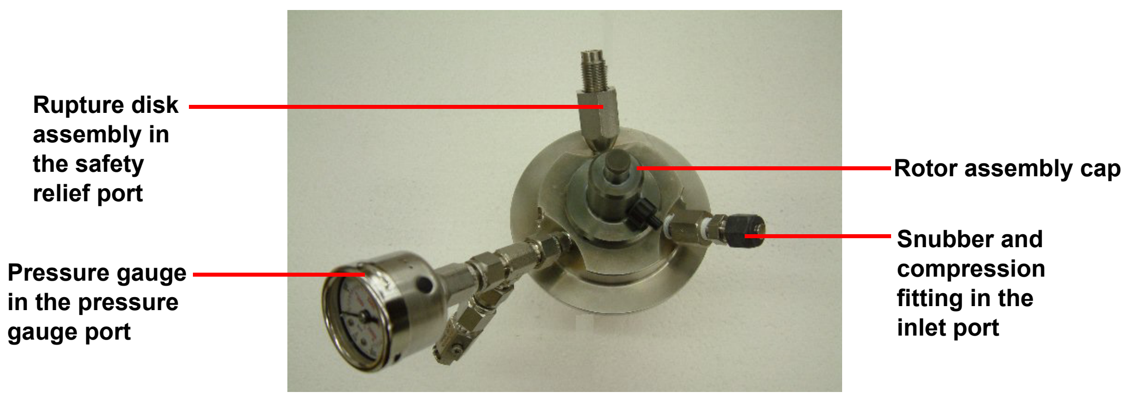


Figure 3 Pressure Cell cup.

The Inlet Port

The inlet port, which is used in the external pressurization mode, is where the compressed gas is introduced to the cup. A pressure manifold is supplied that attaches to the inlet port using a compression connector. A pressure snubber is fitted between the port and the high-pressure line to slow the pressure build and prevent sample from entering the line.

The Pressure Gauge Port

This port is fitted with a pressure gauge which indicates the pressure within the cell, and a relief valve. This valve is only intended to be used when the pressure from the cell cannot be relieved in the usual way (see “Step 6: Pressurizing/Depressurizing the Cell and Running Experiments” on page 30). You will need a 5/8-inch open or box-end wrench to hold the valve body and a 7/16-inch wrench to open the valve.

Figure 4 Pressure gauge port (gauge and valve shown).

Safety Relief Port

The safety relief port is equipped with a rupture disk assembly that is designed to relieve excessive cup pressure. At excessive internal pressure, the rupture disk breaks and propels the internal atmosphere out of the cup.

WARNING: Do not operate the Pressure Cell without the safety relief fitting in place. Do not remove the rupture disc from the safety valve fitting, as this may cause the Pressure Cell to crack during an overpressure condition, resulting in damage and personal injury. The rupture disc should only be replaced by a qualified TA Instruments Service Representative.

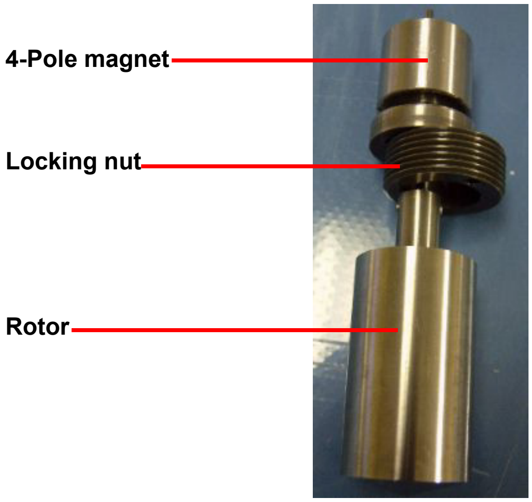
AVERTISSEMENT: N'utilisez pas la cellule à pression si le raccord de sécurité n'est pas installé. Ne retirez pas le disque de rupture du raccord de sécurité, car cela pourrait fissurer la cellule à pression en cas de surpression, entraînant ainsi des dégâts et des blessures corporelles. Le disque de rupture doit être remplacé uniquement par un représentant qualifié du service d'entretien de TA Instruments.

CAUTION: You MUST install the safety relief port with the rupture disk such that it is pointed to rear of the rheometer and away from the operator. This will prevent sample material from being ejected toward the operator in the event of an over-pressure situation.

MISE EN GARDE: Vous DEVEZ installer l'orifice de sécurité avec le disque de rupture de sorte qu'il soit pointé vers l'arrière du rhéomètre et à distance de l'opérateur. Cela permet d'éviter l'éjection de l'échantillon vers l'opérateur en cas de surpression.

Rotor Assembly

The rotor assembly contains the concentric cylinder rotor, which is mounted on a shaft that is radially supported by two sapphire bearings located under the rotor assembly cap. Also attached to the shaft is a four-pole magnet. The rotor assembly installs into the cup using a threaded mount, and seals with a Kalrez® seal. A second Kalrez seal is seated between the cap and the thumbscrew.



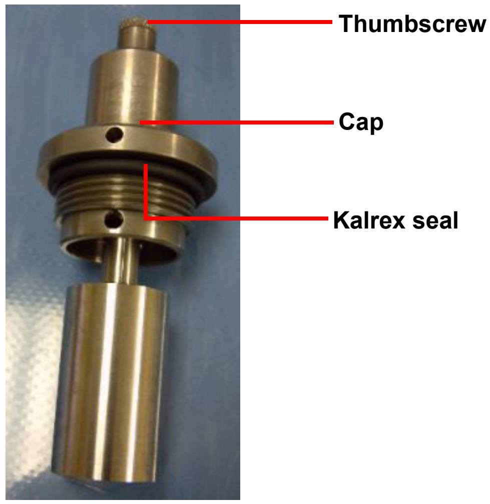


Figure 5 Pressure cell rotor assembly with cap off (left) and on (right).

CAUTION: Prior to use, ensure that the two Kalrez seals are installed and are in good condition. If damaged, replace with seals provided by TA Instruments only.

MISE EN GARDE: Avant de l'utiliser,, assurez-vous que les deux joints d'étanchéité Kalrez sont installés et en bon état. S'ils sont endommagés, remplacez-les par des joints d'étanchéité fournies par TA Instruments uniquement.

Additional Rotors Available

The pressure cell is supplied with a conical end rotor. Other rotors are available: starch (impellor) and vane.

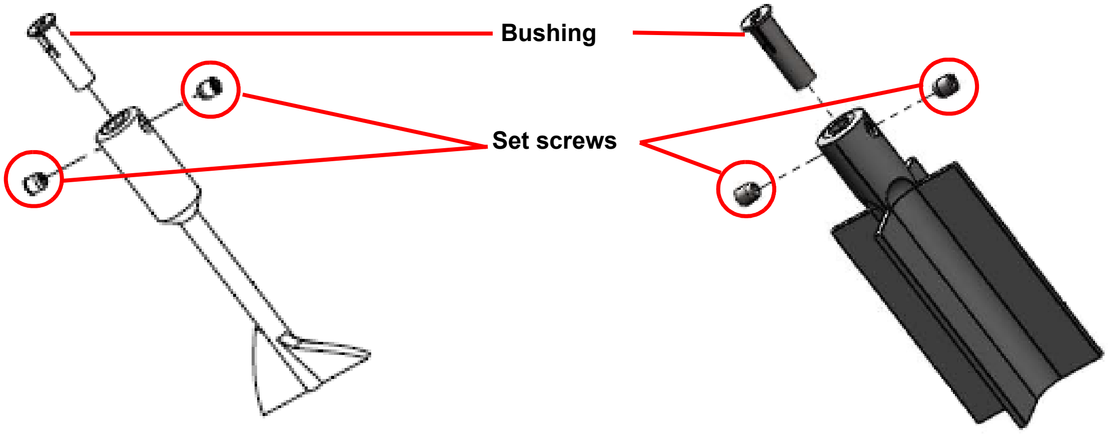


Figure 6 Starch rotor (left), Vane rotor (right).

To change rotors, follow “Disassembling the Rotor” on page 37, and then “Reassembling the Rotor” on page 38 using the new rotor.

Magnet Assembly

The magnet assembly attaches to the rheometer's rotating spindle, and then lowers over the rotor assembly. The spindle collar of the magnet assembly includes an insert adapter. The adapter insert should remain in the collar for use with the AR 2000ex Rheometers. If using the Pressure Cell with a DHR or AR-G2 rheometer, take the adapter insert out of the spindle collar by removing the two Phillips (or cross head) screws extending for the outer surface of the spindle collar. See the figure below.

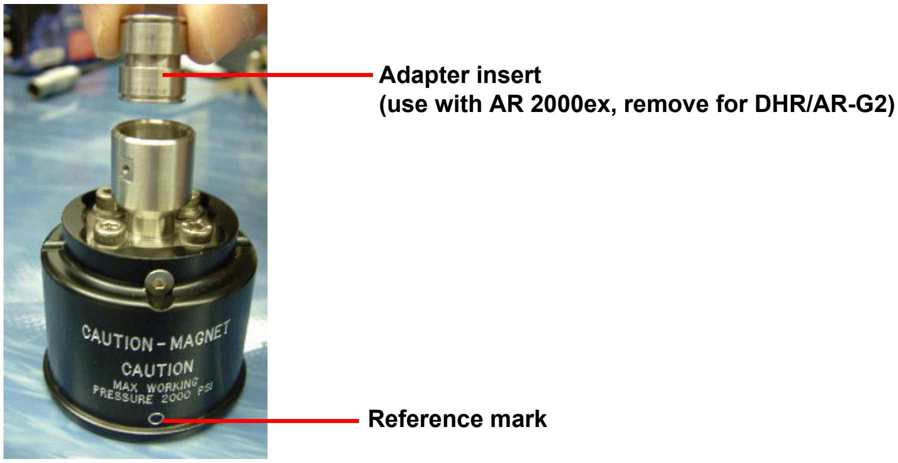


Figure 7 Magnet assembly

Like the rotor assembly, the magnet assembly contains a 4-pole magnet. When the spindle and magnet assembly are rotated, the attraction between the two 4-pole magnets produces a corresponding rotation of the rotor. There is no physical contact between the two assemblies.

CAUTION: Do not place magnetic storage media near the magnet assembly, as it contains a powerful magnet capable of destroying magnetically recorded material.

MISE EN GARDE: Ne placez pas le support de stockage magnétique près de l'ensemble de l'aimant, car il contient un aimant puissant capable de détruire magnétiquement le matériel enregistré.

Pressure Manifold

The Pressure Cell includes a high-pressure manifold assembly that is connected to the rheometer frame. The rigid piping pressure manifold provides strain relief between the Pressure Cell and external high-pressure connections. It also includes necessary valves and gauges for safely pressurizing and depressurizing the cell. It is a critical part of the Pressure Cell assembly and the Pressure Cell should not be operated without the manifold in place. The pressure manifold, shown in the figure below, includes the following:

- Mounting plate and Sorbothane block. The Sorbothane block is a flexible material that provides flex between the rigid Pressure Cell piping assembly and the rheometer frame.
- 1/8-inch and 1/4-inch female NPT fittings for high-pressure connections
- Three way valve for pressurizing, maintaining cell pressure, and depressurizing the cell

- Pressure gauge for monitoring pressure in the cell

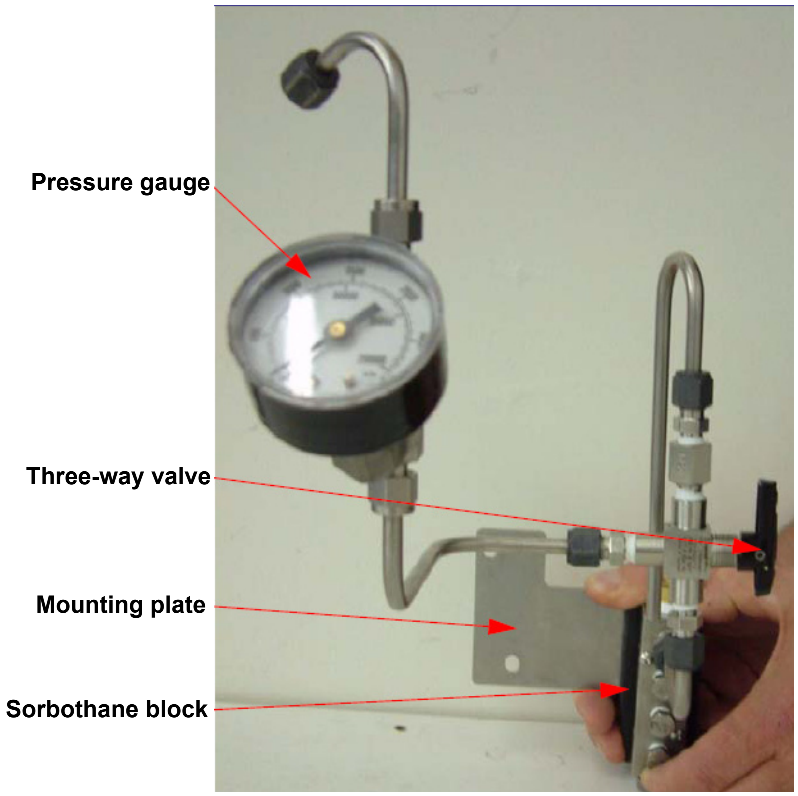


Figure 8 Pressure manifold assembly.

Requirements for External Pressure Source

Fittings are provided for external pressurization up to 138 bar (2000 psi). A high-pressure source must be supplied with 1/8-inch or 1/4-inch NPT male fittings for connection to the manifold supplied by TA Instruments. In addition, a means of isolating the source from the manifold, and of relieving the pressure in the line from the source to the manifold should be provided. The figure below shows a typical set up for external pressurization.

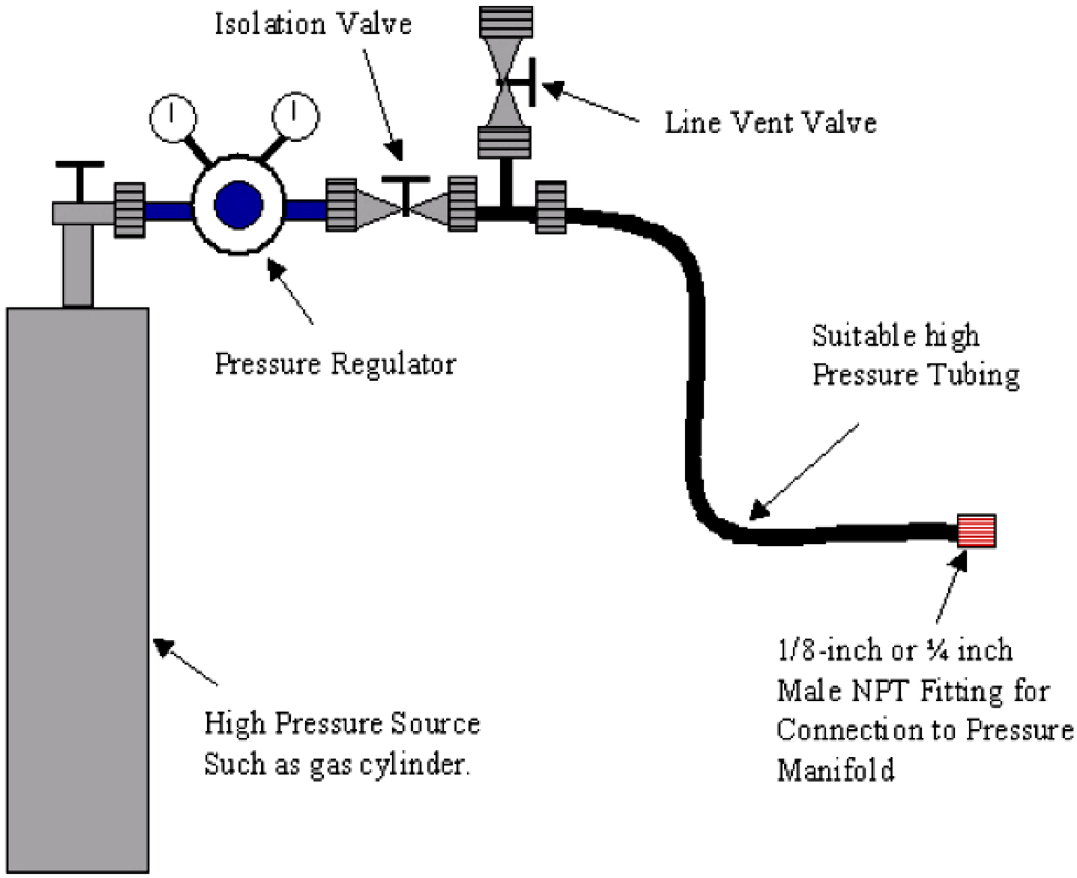


Figure 9 Typical setup for external pressurization.

WARNING: Only use TA Instruments' high-pressure manifold when operating the Pressure Cell. Ensure that the manifold can be isolated from the high-pressure source provided by the user, and that there is a pressure vent valve in the line between the source and the manifold.

AVERTISSEMENT: Utilisez uniquement le collecteur haute pression fourni TA Instruments lors de l'utilisation de la cellule à pression. Assurez-vous que le collecteur peut être isolé de la source haute pression fournie par l'utilisateur et qu'il existe une soupape d'évacuation de la pression dans la conduite entre la source et le collecteur.

Specifications

Table 1: Pressure Cell Operating Specifications

Item/Area	**Specifications**
Stator inner radius	14.00 mm
Rotor outer radius:	13.00 mm
Cylinder immersed height	44.00 mm
Gap	3500 µm (recommended)
Backoff distance	3500 µm
Geometry inertia	67.00 µN.m.s2 (approximate)
Sample volume	9.5 ± 0.5 mL
Temperature range	–10 to 150°C
Maximum applied pressure	138 bar (2000 psi)
Maximum pressure (self-pressurizing)	5 bar (72.5 psi)
Torque range	About 100 µN.m to 0.2 N.m
Maximum angular velocity	50 rad/s
Seal construction	DuPont Kalrez®

Table 2: Pressure Cell Safety Specifications

Item/Area	**Specifications**
Over pressure rupture disk	172 bar (2500 psi)
Hydraulically tested to	414 bar (6000 psi)

Operational Limits

CAUTION: To prevent sample entering the upper part of the cell and contaminating the bearings, the cell should not be used above the limits given below. Exceeding these limits may also cause mechanical damage to the cell.

MISE EN GARDE: Pour éviter que l'échantillon pénètre dans la partie supérieure de la cellule et contamine les paliers, la cellule ne doit pas être utilisée au-delà des limites indiquées ci-dessous. Dépasser ces limites peut également provoquer des dégâts mécaniques à la cellule.

Table 3: Pressure Cell Operational Limits

Item/Area	**Specifications**
Maximum angular velocity	50 rad/s
Maximum sample viscosity	The geometry should not be forced into the sample. Light hand pressure should be all that is required.
Maximum frequency	50 Hz (314 rad/s)

NOTE: The quality of data obtained using the Pressure Cell cannot be expected to match that obtained when conventional measuring systems are used with the rheometer. Some of the normal calibration routines are not relevant to, or cannot be used with the Pressure Cell. Alternative calibration routines are described in this manual.

Chapter 2:

Installing and Setting Up the Pressure Cell

Overview

The Pressure Cell is shipped with the rotor installed in the Pressure Cell cup, and the high-pressure piping manifold fully assembled for use with the DHR Series Rheometers. Installing the Pressure Cell from the initial shipped configuration requires some disassembly. Disassembly instructions are as follows:

Step 1: Unpack the preassembled Pressure Cell cup and rotor. Using the spanner (Tommy bar) provided, remove the rotor assembly from the Pressure Cell cup, and carefully place the rotor in a safe location. Remove foam packing material from Cup.

NOTE: All the compression fittings that are used on the manifold assembly have a specific tightening procedure for the first time the nut is tightened on the tubing during the manufacturing process. Subsequent disassembly and remake of any fitting should be done by first putting a reference mark on the nut and the body of the fitting before loosening the fitting. After reassembly of the fitting to a finger tight condition, the nut should then be tightened with a wrench so that the mark on the nut aligns with the mark on the body of the fitting. A second wrench should be used to hold the body of the fitting in place while turning the nut.

Step 2: Unpack the preassembled piping manifold assembly. Using a wrench, remove the furthest extending angled pipe (shown in Figure 10 below) from the Tee fitting joining the gas inlet and pressure gauge.

Step 3 (for AR Series use only): To use the Pressure Cell with AR Series Rheometers, replace the long pipe on the manifold assembly (identified below) with the shorter pipe in the kit. Refer to Figure 11 below to see the manifold assembled for AR use.

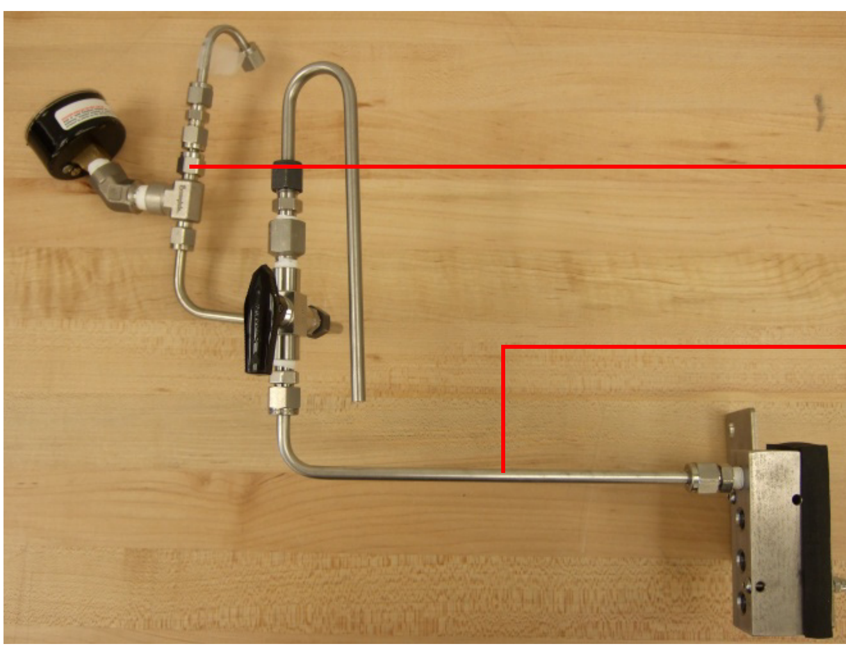


Figure 10 Manifold assembly.

The figure below shows the fully disassembled configuration. Note that the manifold below shows the shorter pipe for AR Series use.

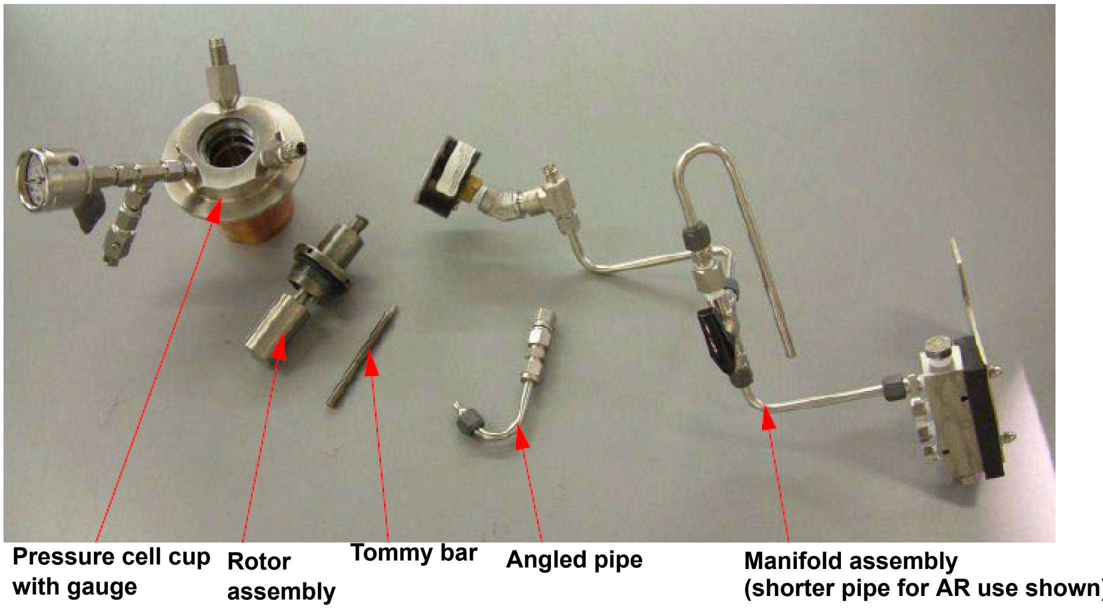


Figure 11 Entire Pressure Cell configuration disassembled.

The steps on the next several page provide the instructions needed to install and use the Pressure Cell.

Installing the Pressure Cell

Depending upon your instrument, see "Step 1: Install High-Pressure Piping Manifold (for DHR Series)" below or "Step 1: Install High-Pressure Piping Manifold (for AR Series)" on page 23.

Step 1: Install High-Pressure Piping Manifold (for DHR Series)

1 Fit the adapter bracket to the back of the instrument using the screws and washers provided.

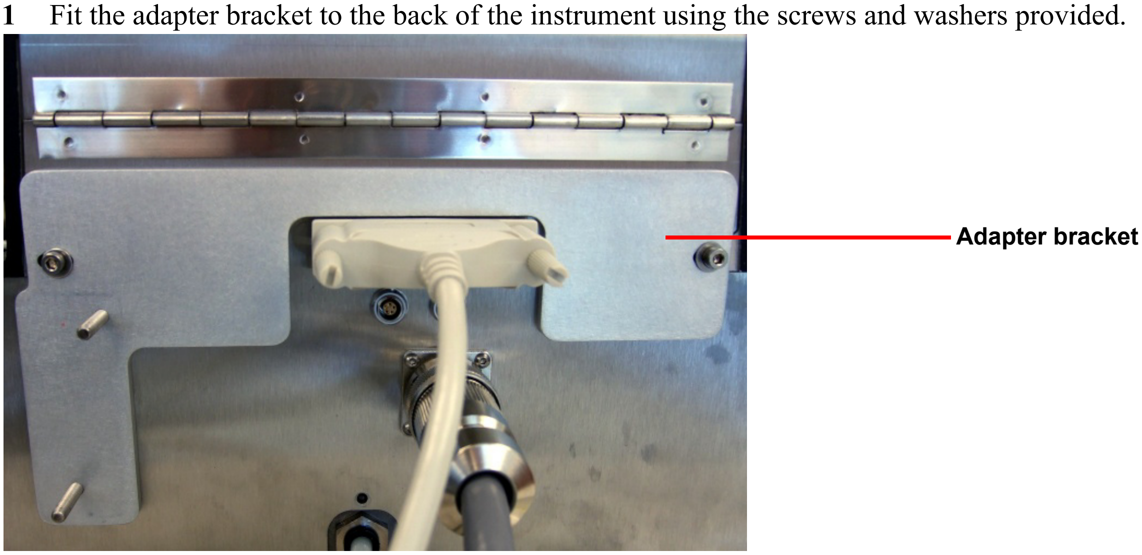


Figure 12 Adapter bracket installed on back on instrument.

2 Fit the Sorbothane block over the two threaded studs on the adapter bracket.

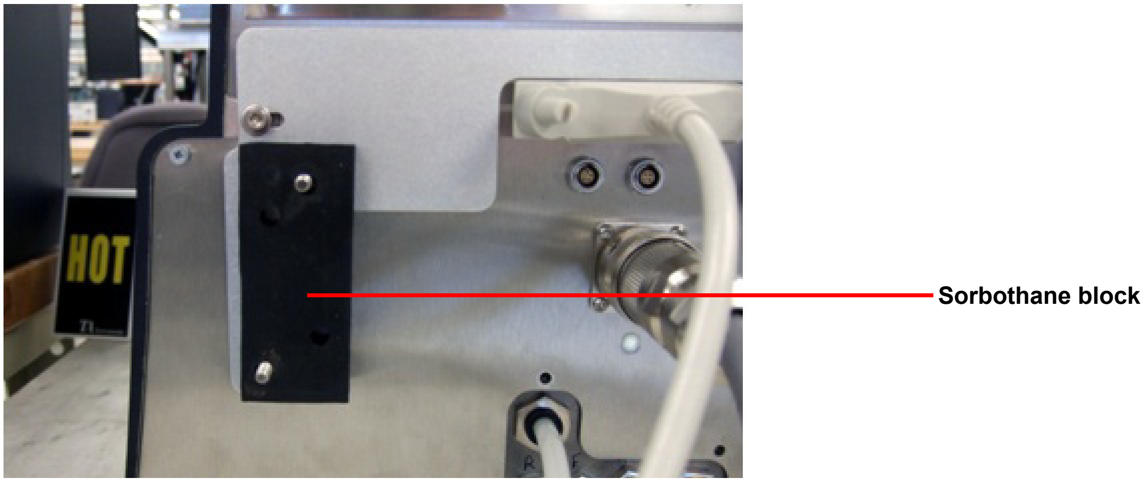


Figure 13 Sorbothane block installed over adapter bracket.

3 Attach the manifold bracket to the adapter bracket using the thumbscrews.

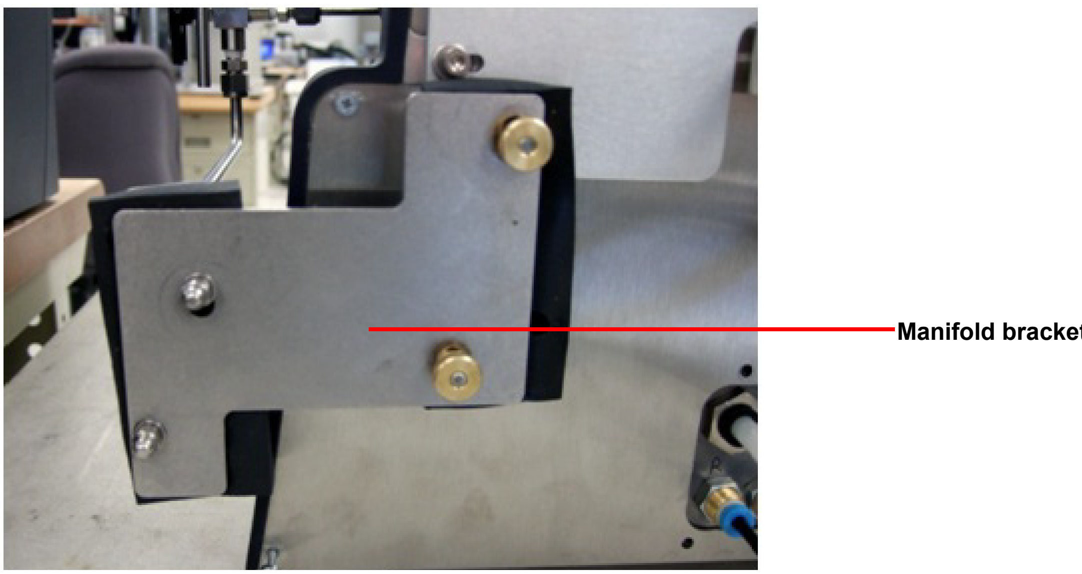


Figure 14 Manifold bracket installed on adapter bracket.

Step 1: Install High-Pressure Piping Manifold (for AR Series)

The pressure manifold attaches to the lower right rear as viewed from front of rheometer or left lower corner when facing rear of the AR Rheometer, as circled below.

Figure 15 Manifold location on back of rheometer.

Attach the mounting plate and Sorbothane block to the two M5 using the cap head screws provided, as shown below.

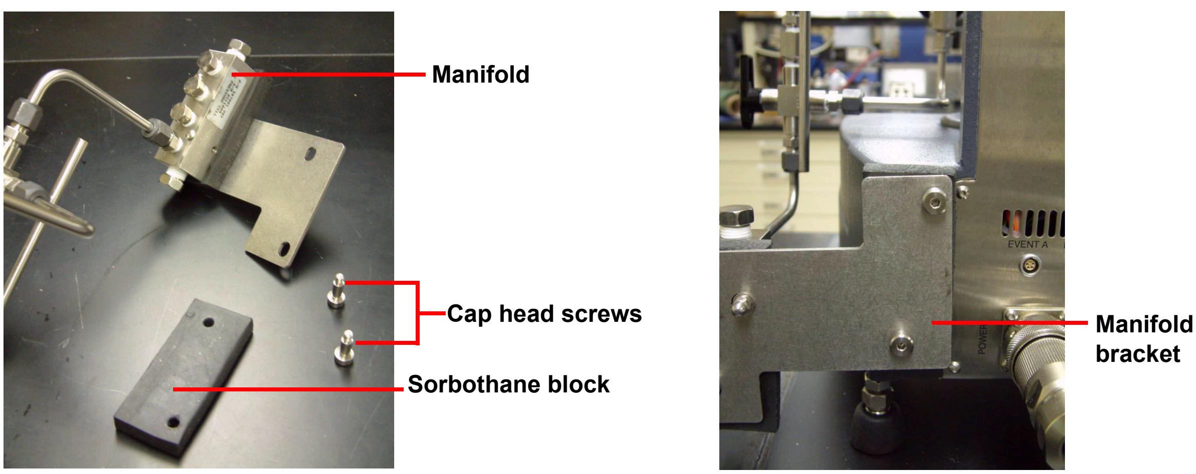


Figure 16 Manifold installed.

Step 2: Install and Configure Pressure Cell Cup and Rotor

Follow the instructions below to both install and configure the Pressure Cell Cup and rotor:

1 Locate the Peltier Concentric Cylinder jacket. Remove the Peltier jacket, if it is installed on the rheometer.

2 Remove any Peltier Concentric Cylinder cups and remove the two knurled screws that fasten the standard Peltier cups in place (note the standard knurled screws can not be used with the Pressure Cell).

3 Insert the Pressure Cell cup into the jacket with the safety relief valve facing to the rear of the cup. Note the "Hot" symbol and white Smart Swap alignment line markings on the front of the Peltier jacket. Fix the cup in position in the jacket using the two hex head screws and hex keys provided with the Pressure Cell. See the figure below.

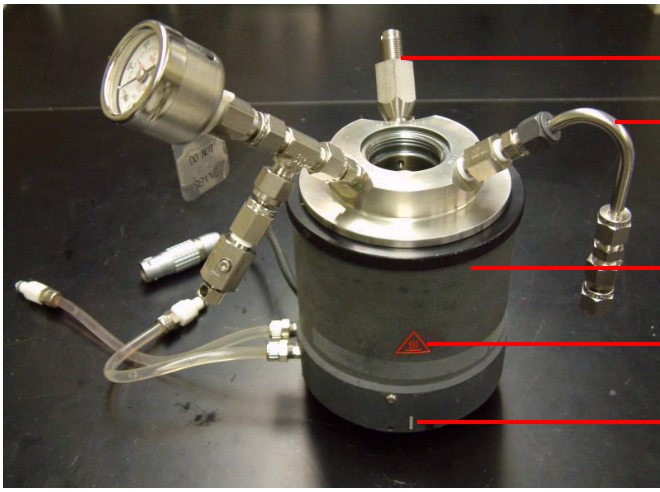


Figure 17 Pressure Cell cup installed in Peltier jacket.

4 Connect angled high-pressure pipe to the cell inlet port, ensuring that the straight part of the pipe is vertical, as shown in Figure 17. Tighten the compression connector as directed in the NOTE on page 20.

5 Locate the rotor assembly. Place the small magnet onto the rotor assembly, as shown below, such that the small magnet is vertically aligned with the scribe line etched on the collar on the rotor assembly.

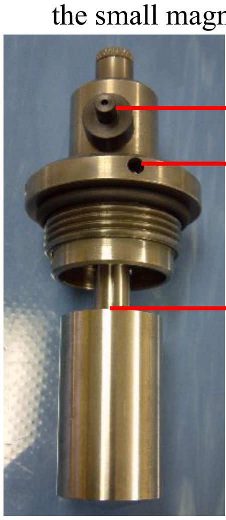


Figure 18 Rotor assembly with small magnet.

6 Hand-tighten the rotor assembly into the cup. Fully tighten the rotor until flush with the cup using the wrench (also called a Tommy bar).

7 Position the small magnet on the rotor to face in the front center of the Peltier jacket as shown below.

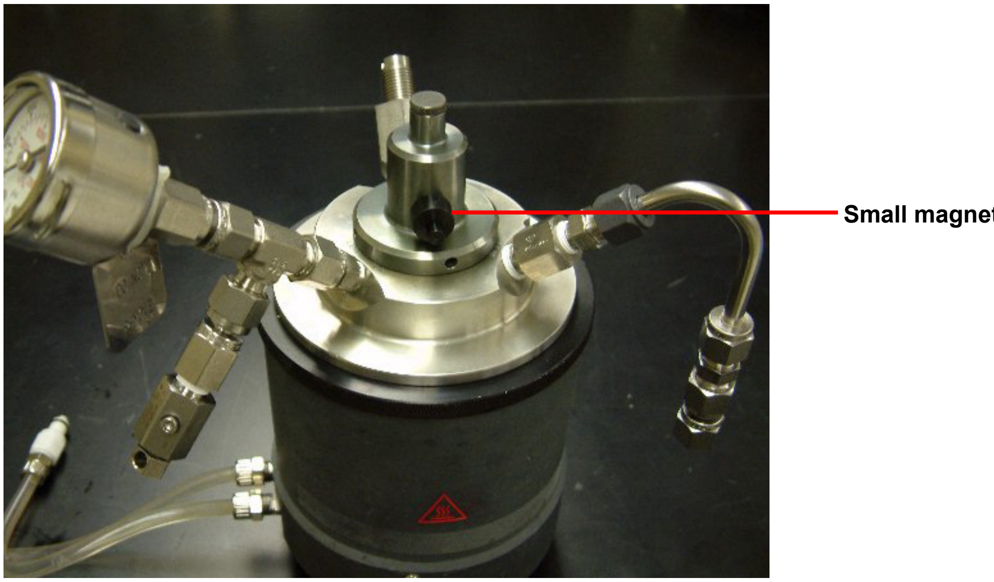


Figure 19 Orientation of small magnet.

8 Install Peltier jacket on rheometer using Smart Swap connectors.

9 Install the magnet assembly onto the shaft of the rheometer.

10 Rotate the draw rod so the magnet assembly reference mark is aligned with the small magnet on the Rotor. Ensure that the reference mark on the upper geometry remains aligned with the small magnet by lightly holding the rheometer draw rod and begin lowering the rheometer head as shown below.

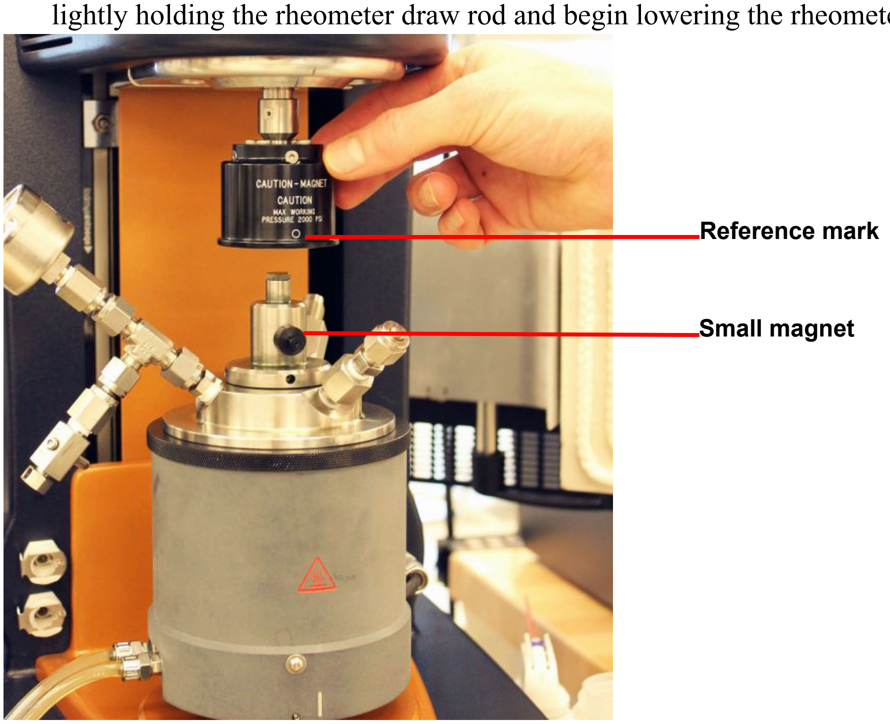


Figure 20 Aligning the reference mark.

11 At a gap of about 20 mm between the shoulder on the rotor assembly and the underside of the upper magnet assembly, the magnets in the upper assembly will engage with those in the rotor assembly as shown below. A small noise will be heard when this happens and a change of a few Newtons will be seen in the normal force reading. Immediately stop moving the rotor assembly down and remove the small magnet.

Figure 21 Magnets engaging.

Step 3: Positioning Gap and Pressure Cell Calibrations

Once the Pressure Cell cup and rotor have been installed, you will need to position the gap and perform the calibrations as directed in this section.

CAUTION: The standard calibration routines used by TRIOS software for zero gap, geometry inertia, and bearing friction are not suitable for use with the Pressure Cell. When the Pressure Cell is selected as the measuring geometry, these routines are either disabled or are replaced by more appropriate routines. Do not attempt to use or calibrate the Pressure Cell unless this geometry is selected.

MISE EN GARDE: Les programmes d'étalonnage standard utilisés par le logiciel TRIOS pour l'écartement zéro, l'inertie de la géométrie et le frottement des coussinets ne sont pas appropriées pour la cellule à pression. Lorsque la cellule à pression est sélectionnée comme géométrie de mesure, ces programmes sont désactivés ou remplacés par des programmes plus appropriés. N'essayez pas d'utiliser ou d'étalonner la cellule à pression à moins de sélectionner cette géométrie.

1 Ensuring that the Pressure Cell geometry is selected in the software, engage the magnetic coupling and manually position the head so that the gap between the coupling and the top of the cell is about 2 mm before running the zero gap routine. Then go to the operating gap of 3500 µm.

2 When the Pressure Cell is the selected geometry on an AR2000ex, the Gap Zero Mode of normal force with a value of 5N will be used. This will override any other settings in TRIOS software.

3 Conduct the **Bearing Friction Calibration**. The bearing friction routine used when the Pressure Cell is selected as the geometry is slightly different from the standard routine. The calibration should be conducted at a geometry gap of 3500 µm. The bearing friction calibration must be done again when another measuring system is used. A typical value for the Pressure Cell should be between 8 and 15 µN.m / (rad/s). This is about ten times higher than for other geometries.

4 Map the air bearing. Perform a **Rotational Mapping** at a gap of 3500 µm using the standard mapping routine.

NOTE: DO NOT USE PRECISION MAPPING ROUTINES WITH THE PRESSURE CELL.

CAUTION: It is important that the bearing is re-mapped before any other measuring system is used.

MISE EN GARDE: Il est important de mapper à nouveau le coussinet avant d'utiliser un autre système de mesure.

NOTE: When changing to other geometries, TRIOS Software does not restore the previous settings. However, the mapping table is cleared and the bearing friction is reset to zero. Any functions that were previously unavailable are reactivated and the gap zero mode settings are restored, because the settings were not overwritten.

5 Check the cell by running peak-hold test at 0.05 rad/s and a duration time of 126 sec. The peak-to-peak residual torque should not be larger than 100 µN.m.

Step 4: Loading a Sample

Samples are loaded in the Pressure Cell after the cell is set up and calibrated. The following steps will detail the sample loading procedure.

1 Rotate the drawrod so the reference mark on the magnet assembly is facing the front of the instrument. Raise the rheometer head high enough to place the small magnet on the rotor. Once the small magnet is in place, raise the rheometer head to the maximum height.

NOTE: DO NOT REMOVE MAGNET ASSEMBLY FROM THE RHEOMETER HEAD. IF IT IS REMOVED, THE MAPPING WILL NO LONGER BE AS EFFECTIVE, CAUSING AN INCREASE IN RESIDUAL TORQUE.

2 Remove the Peltier jacket from rheometer.

3 Leaving the small magnet in place, gently remove the rotor from the cup.

4 Load the sample into the cup. For very viscous samples, you may find it easiest to weigh the sample in the cup, if the sample density is known (this can be done after removing the cup from the jacket).

NOTE: Volume is 9.5 ± 0.5 mL.

5 Ensure that the small magnet is still aligned with the mark on the rotor assembly.

6 Replace the rotor assembly and fully tighten.

7 Replace the Peltier jacket onto the Smart Swap base of the rheometer.

8 Rotate the draw rod so the magnet assembly reference mark is aligned with the small magnet on the Rotor. Ensure that the reference mark on the upper geometry remains aligned with the small magnet by lightly holding the rheometer draw rod and begin lowering the rheometer head as shown in Figure 20.

9 At a gap of about 20 mm between the shoulder on the rotor assembly and the underside of the upper magnet assembly, the magnets in the upper assembly will engage with those in the rotor assembly as shown in Figure 21. A small noise will be heard when this happens and a change of a few Newtons will be seen in the normal force reading. Immediately stop moving the rotor assembly down and remove the small magnet.

10 Lower the instrument head to the geometry gap (default 3500 µm). Do not zero the gap.

Step 5: Align Manifold and Make Manifold Connections

After the sample is loaded, you will need to align the manifold as follows. The pressure manifold should only be connected and disconnected from the cup assembly at the bottom of the angled pipe connected to the cup as shown below. Please refer to the NOTE on page 20.

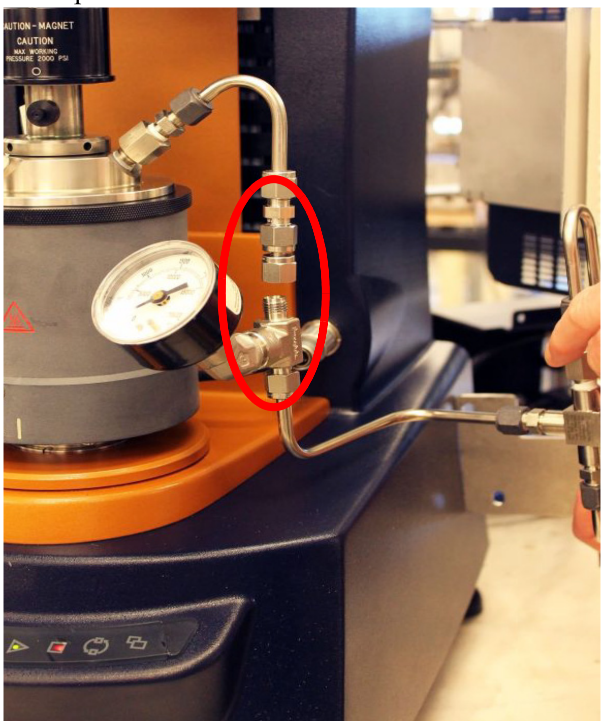


Figure 22 Location for connecting/disconnecting manifold.

1 Prior to connecting the pressure manifold to the angled pipe, slacken off to finger tight the compression connectors on the manifold in order to easily align the manifold with the pipe mounted on the cup. DO NOT USE EXCESSIVE FORCE TO POSITION THE PRESSURE MANIFOLD.

2 Finger-tighten ONLY the manifold to the angled pipe mounted on the cup, using the connector indicated in the figure below.

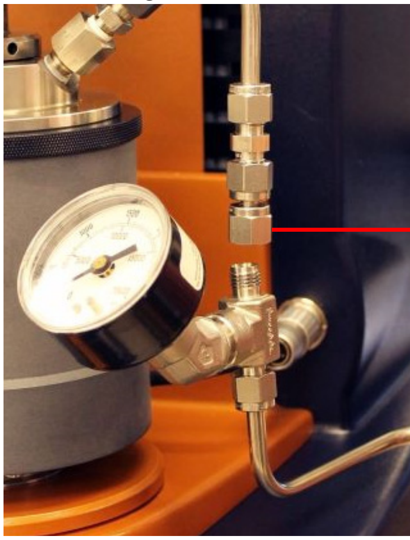


Figure 23 Connector for connecting/disconnecting manifold.

3 Once this fitting is finger tight, fully tighten all other compression fittings as directed in the NOTE on page 20.

4 Finally, fully tighten the angled pipe to the manifold as directed in the NOTE on page 20.

CAUTION: To avoid putting excessive force on the Pressure Cell, make sure that connection or disconnection between the Pressure Cell and the manifold is made at the breakage point compression connector only (see Figure 22 and Figure 23. Connection or disconnection should be made at no other point while the Pressure Cell is mounted on the rheometer).

MISE EN GARDE: Pour éviter d'exercer une force excessive sur la cellule à pression, assurez-vous que la connexion et la déconnexion entre la cellule à pression et le collecteur se fait uniquement au niveau du connecteur de compression du point de rupture (voir Figure 22 et Figure 23). La connexion et la déconnexion ne doit pas se faire à aucun autre point lorsque la cellule à pression est montée sur le rhéomètre).

NOTE: To disconnect the Pressure Cell from the manifold, ensure that both the cell and the manifold are depressurized. Then disconnect the compression connector indicated in Figures 9.20 and 9.21.

WARNING: Before disconnecting the Pressure Cell from the manifold, ensure that neither the cell nor the manifold is pressurized, and that both are cool enough to touch.

AVERTISSEMENT: Avant de déconnecter la cellule à pression du collecteur, assurez-vous que ni la cellule ni le collecteur n'est sous pression et qu'ils sont tous les deux assez froids pour être touchés.

Step 6: Pressurizing/Depressurizing the Cell and Running Experiments

When the cell is fully installed, it can be operated in either self-pressurization mode, in which the pressure is due to the volatility of the sample, or in external pressurization mode, in which the pressure is provided by an external source. The pressure achieved when the cell is used in self-pressurizing mode will depend on the sample and temperature. As a guide, the vapor pressure of water at 150°C is about 4.76 bar (69 psi.).

When the cell has been assembled correctly as directed in this chapter, it can be pressurized. The manifold contains a three-way valve. The three positions for the valve are shown below.

Vertical Up—Pressure Relief

Connection between the Pressure Cell and atmosphere.

Vertical Down—Pressure Build (external pressurization mode)

Connection between the Pressure Cell and the connector block on the manifold.

Horizontal—Off

Isolation between the Pressure Cell and the connector block on the manifold.

Figure 24 Three-way valve positions.

Chapter 3:

Pressure Cell Use and Maintenance

Running Experiments in Self-Pressurization Mode

After the manifold is installed, all fittings have been tightened, and the sample has been loaded, follow these steps to run an experiment in the self-pressurization mode.

1 Make sure the three-way valve on the manifold is set to the OFF position (horizontal).

NOTE: The secondary pressure relief valve on the pressure gauge port should only be opened if the primary pressure relief passage becomes clogged. If this valve was opened, make sure it is closed.

2 Set the testing temperature from the software.

3 Program the test procedure.

4 Run the experiment.

5 After running the experiment, depressurize the cell by following these steps:

- **a** Ensure the Pressure Cell is isolated from the high pressure source.
- **b** When the experiment is complete, the temperature should be set back to 25°C (ambient temperature) before attempting to relieve the pressure in the cell.
- **c** Once the cell has cooled, set the three-way valve on the manifold to the “Pressure Relief” position (arrow pointing upward). Note that the pressure may reduce very slowly, due to the snubbers in the manifold.

NOTE: There are two snubbers in the manifold, one of which is located on the cell itself and the other in the vent line, as described earlier in this chapter. These are used to slow the build and relief of pressure in or from the cell. The snubbers may become clogged with sample, preventing the cell pressure from being properly relieved (this problem is more likely to occur when external pressurization is being used). You must maintain the cleanliness of the snubbers, ensuring that gas can pass through them. You can verify that they are operating properly by pressurizing and depressurizing the cell without a sample. If there is any doubt concerning the cleanliness or operation of the snubbers, they should be replaced.

NOTE: The pressure relief valve on the cell’s pressure gauge port can also be used to relieve the pressure in the cell. This should only be done if the three-way valve on the manifold cannot be used, if, for example, the snubbers have become blocked. The cell should also be at room temperature before using this valve, and appropriate gloves and safety glasses should be worn.

The pressure gauge located on the Pressure Gauge Port and the pressure gauge on the manifold can be used to help verify that the snubbers are not clogged. After relieving the pressure in the cell using the manifold and three-way valve, the two gauges should read zero (0) pressure. If the snubbers are clogged, the pressure gauge in the Pressure Gauge Port may still register pressure. If that condition occurs, use the relief valve on the Pressure Gauge Port to release pressure from the cell.

- Disconnect the Pressure Cell from the manifold at the designated disconnection point at bottom of the angled pipe.
- Raise the head and decouple the magnet assembly and the rotor, and remove the Peltier jacket from the rheometer using the procedures outlined previously in "Step 4: Loading a Sample" on page 27.
- Remove the cup and rotor for cleaning. See "Maintaining the Cell" on page 35 for details.

CAUTION: Although the pressure gauge on the cell may to appear to register zero, the level of pressure in the cell may still be above ambient.

MISE EN GARDE: Bien que le manomètre de la cellule semble enregistrer zéro, le niveau de pression de la cellule peut toujours être ambiant.

Running Experiments in External Pressurization Mode

NOTE: If the maximum pressure of the external pressure source is greater than 138 bar (2000 psi), (e.g. N2 tank), the source must be regulated to a maximum value of 138 bar (2000 psi).

After the manifold is installed, all fittings have been tightened, and the sample has been loaded, follow these steps to run an experiment in the external pressurization mode.

1 Make sure the three-way valve on the manifold is set to the OFF position (horizontal).

NOTE: The secondary pressure relief valve on the pressure gauge port should only be opened if the primary pressure relief passage becomes clogged. If this valve was opened, make sure it is closed.

WARNING: Before applying high pressure, check for leaks at low pressure. Raise the pressure of the cell, gradually, making frequent leak checks.

AVERTISSEMENT: Avant d'exercer une haute pression, vérifiez s'il y a des fuites à basse pression. Augmentez progressivement la pression de la cellule, en vérifiant régulièrement s'il y a des fuites.

2 Regulate the external pressure source to the pressure required for the experiment. THIS PRESSURE CANNOT EXCEED 138 bar (2000 psi).

3 Gently pressurize the cell by slowly opening the three-way valve on the manifold to the pressure build position (arrow pointing vertically downwards). Check the Pressure Cell to make sure there are no leaks. If it is free from leaks at low pressure, raise the pressure gradually, making frequent leak checks using a liquid leak test material.

4 During operation, the Pressure Cell should be isolated from the high-pressure source. Set the three-way valve on the manifold to OFF (arrow horizontal), and relieve the pressure in the line between the high-pressure source and the connector block. If the cell is free from leaks, the pressure in the cell will be maintained.

WARNING: During operation, isolate the Pressure Cell from the high-pressure source, and relieve the pressure in the line between the source and the manifold.

AVERTISSEMENT: Pendant l"utilisation de l'instrument, isolez la cellule à pression de la source de haute pression et évacuez la pression de la conduite située entre la source et le collecteur.

5 Set the testing temperature from the software.

6 Program the test procedure.

7 Run the experiment.

8 After running the experiment, depressurize the cell by following these steps:

a Ensure the Pressure Cell is isolated from the high pressure source.

b When the experiment is complete, the temperature should be set back to 25°C (ambient temperature) before attempting to relieve the pressure in the cell.

c Once the cell has cooled, set the three-way valve on the manifold to the pressure relief position (arrow pointing upward). Note that the Pressure Cell may reduce very slowly, due to the snubbers in the manifold.

NOTE: There are two snubbers in the manifold, one of which is located on the cell itself and the other in the vent line, as described earlier in this chapter. These are used to slow the build and relief of pressure in or from the cell. The snubbers may become clogged with sample, preventing the cell pressure from being properly relieved (this problem is more likely to occur when external pressurization is being used). You must maintain the cleanliness of the snubbers, ensuring that gas can pass through them. You can verify that they are operating properly by pressurizing and depressurizing the cell without a sample. If there is any doubt concerning the cleanliness or operation of the snubbers, they should be replaced.

NOTE: The pressure relief valve on the pressure gauge port on the Pressure Cell can also be used to relieve the pressure in the cell. This should only be done if the 3-way valve on the manifold cannot be used, if, for example, the snubbers have become blocked. The cell should also be at room temperature before using this valve, and appropriate gloves and safety glasses should be worn.

The pressure gauge located on the pressure gauge port and the pressure gauge on the manifold can be used to help verify that the snubbers are not clogged. After relieving the pressure in the cell using the manifold and three-way valve, the two gauges should read zero (0) pressure. If the snubbers are clogged, the pressure gauge in the pressure gauge port may still register pressure. If that condition occurs, use the relief valve on the pressure gauge port to release pressure from the cell.

- Disconnect the Pressure Cell from the manifold at the designated disconnection point at bottom of the angled pipe.
- Raise the head and decouple the magnet assembly and the rotor, and remove the Peltier jacket from the rheometer using the procedures outlined previously in “Step 4: Loading a Sample” on page 27.
- Remove the cup and rotor for cleaning. See “Maintaining the Cell” on page 35 for details.

CAUTION: Although the pressure gauge on the cell may to appear to register zero, the level of pressure in the cell may still be above ambient.

MISE EN GARDE: Bien que le manomètre de la cellule semble enregistrer zéro, le niveau de pression de la cellule peut toujours être ambiant.

Maintaining the Cell

WARNING: Before removing the heating jacket from the rheometer or the cell from the jacket, ensure that cell and manifold are not under pressure, and that the both are at safe touching temperature.

AVERTISSEMENT: Avant de retirer la chemise de réchauffage du rhéomètre ou la cellule de la chemise, assurez-vous que la cellule et le collecteur ne sont pas sous pression et que les deux sont à une température de contact sécurisée.

Cleaning the Pressure Cell Cup

The cup can be cleaned simply by washing it in solvent or other type of cleaner (water, etc.), depending on the material being test, after removal from the heating jacket.

Cleaning the Rotor Assembly

CAUTION: When cleaning the rotor, observe the following precautions:

(1) Do not apply a sideways force to the rotor. Doing so can bend, break, or otherwise damage it. If a bent rotor shaft is suspected (will not rotate freely), hold the rotor assembly horizontally by the upper body (do not hold by the rotor), and spin the rotor while observing the edge. No discernible wobble should be seen as the edge of the rotor rotates.

(2) Do not allow sample or solvent to flow upward into the upper portion of the body of the rotor assembly, as this may result in a change in the friction of the sapphire bearings.

MISE EN GARDE: Lors du nettoyage du rotor, respectez les mesures de précaution suivantes:

(1) N'exercez pas de force latérale sur le rotor. Cela risque de plier, casser ou d'endommager le rotor. Si vous suspectez qu'un arbre de rotor est plié (il ne tourne pas librement), tenez l'ensemble du rotor horizontalement par la partie supérieure (ne le tenez pas par le rotor) et faites tourner le rotor tout en observant le bord. Il ne doit y avoir aucun déséquilibre visible lorsque l'extrémité du rotor tourne.
(2) Ne laissez pas l'échantillon ou le solvant s'écouler vers le haut dans la partie supérieure du corps du rotor, car cela risque de modifier le frottement des paliers en saphir.

If sample fluid has flowed into the sapphire bearings and magnet of the rotor assembly, the rotor may become "sticky" (not rotate freely). In this case, the rotor assembly must be dismantled and cleaned as follows:

1. Hold the rotor assembly by the upper portion only. Be careful not to bend the rotor shaft—see Caution #1 above.
2. Place the tips (bent at 90°) of the supplied pliers into the holes of the locking nut and unscrew the nut by rotating counterclockwise (anticlockwise).
3. Remove all inner components by gently pulling the rotor straight out of the assembly. Clean the inner components using an appropriate solvent. Cellophane tape can be used to clean surfaces that have been contaminated by solid particles, but be certain that no adhesive residue remains on the components.

4 If sample has flowed to the upper sapphire bearing, remove the thumbscrew and clean the inner components using solvent.

5 Reassemble the pieces in the reverse order, being careful not to overtighten the locking nut (hand-tight is sufficient).

NOTE: Typically, sapphire bearings are designed to work **"**dry.**"** The Pressure Cell is therefore supplied without lubricant. However, it has been found that a small amount of suitably compatible grease applied to the bearings can reduce the risk of a "sticky" bearing following contamination of the bearing by condensation or overflow of sample fluid.

Disassembling the Rotor

1 Insert the wrench (also called a Tommy Bar) provided into one of the six holes in the rotor assembly cap and turn the rotor assembly counterclockwise until the entire assembly can be removed.

2 Place the rotor assembly upside down on a flat surface (table or lab bench). Be careful not to bend the rotor shaft—see Caution #1 on the previous page.

3 Place 90° pliers into the holes of the locking nut and unscrew the locking nut by rotating it counterclockwise (anticlockwise). See the figure below. (You may find it easier to hold the locking nut steady with the pliers and rotate the rotor cap clockwise.)

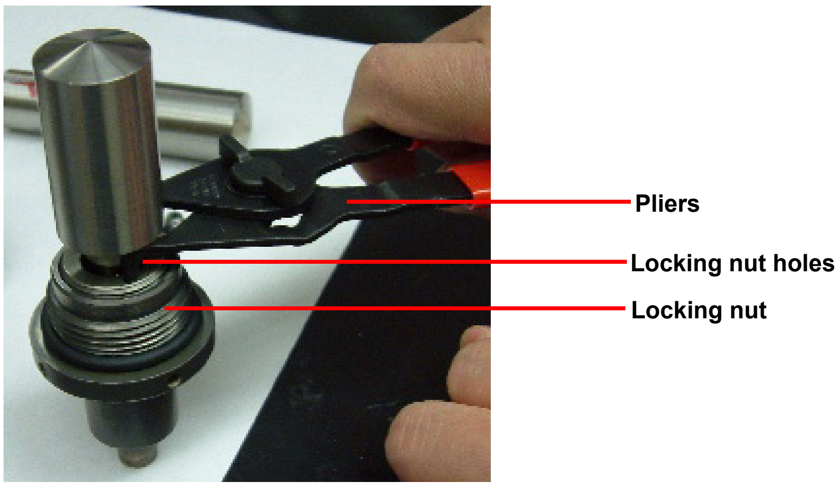


Figure 25 Taking apart the rotor cell assembly.

4 Remove the cap covering the magnet (see the figure below for reference, if needed).

5 Before disassembling the remaining parts, note the scribe lines on the magnet and bob collar. This will allow you to realign the magnet properly after cleaning. See the figure below.

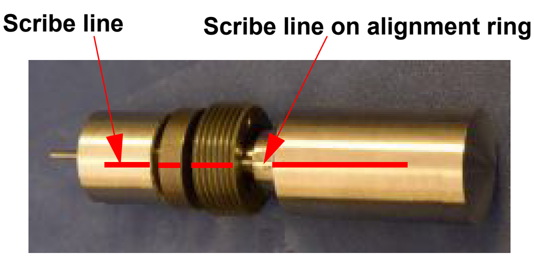


Figure 26 Marking magnet to line up with alignment hole.

6 Continue to remove parts only if you have a torque wrench to retighten the rotor setscrews again as directed in step 13 on the next page.

7 Loosen the two setscrews on the rotor to remove the rotor from the shaft. The locking nut and sapphire bearing holder can now be removed from the shaft. See the figure below for reference.

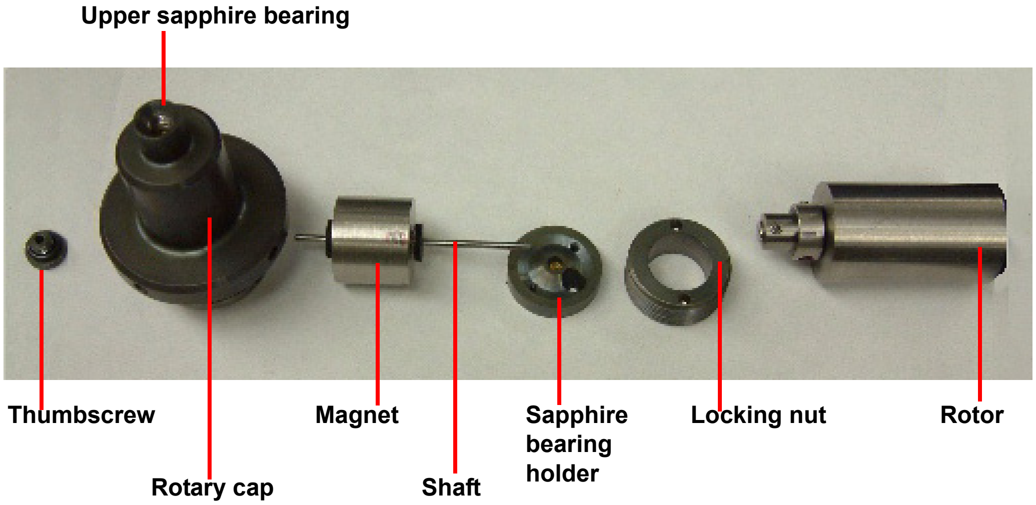


Figure 27 Parts of the rotary assembly (in correct order of assembly).

8 Clean the inner components using a hydro-carbon based solvent (e.g., Alcohol, Heptane).

CAUTION: DO NOT use Acetone. Acetone that gets into the magnet/shaft assembly will degrade the epoxy, causing the shaft to break away from the bushing.

MISE EN GARDE: N'utilisez PAS de l'acétone. L'acétone qui pénètre dans l'ensemble de l'aimant/ l'arbre détériore l'époxy, provoquant le détachement de l'arbre de la douille.

Cellophane tape can be used to clean magnet surfaces that have been contaminated by solid particles, but be certain that no adhesive residue remains on the components.

9 If sample has flowed to the upper sapphire bearing, remove the thumbscrew and clean the inner components using solvent. A pipe cleaner can be used to clean inside surfaces.

Reassembling the Rotor

Make sure all of the parts are dry after the cleaning process and follow the steps below to reassemble the pieces.

10 Slide the sapphire bearing holder onto the magnet shaft making sure the flat side is facing towards the rotor (which will be on the bottom).

11 Slide the locking nut over the shaft with the plier holes facing towards the rotor.

12 Align the magnet's mark with the rotor alignment ring, insert the shaft into the rotor, and make sure the shaft bottoms out in the rotor.

13 Tighten the two setscrews on the rotor, using a torque wrench to adjust the setscrews to 8.5 lbf-in or .96 N-m.

14 Turn the rotary cap upside down and rest it on a flat surface (table or lab bench).

15 Taking the parts already loosely assembled, insert the shaft into the rotary cap until you feel it hit the bottom. You may need to move it around slightly until it drops all the way through. The tip of the shaft should be visible at the top of the rotor as seen in the figure below.

Figure 28 Tip of shaft visible.

16 Using the 90° pliers, screw down the locking nut by rotating it clockwise. See the figure below. You may find it easier to tighten the nut by rotating the rotary cap counterclockwise and holding the locking nut steady with the pliers.

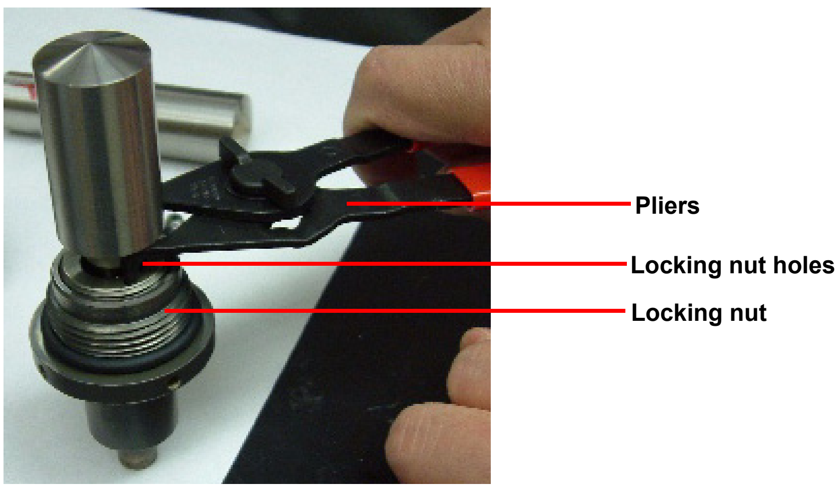


Figure 29 Taking apart the rotor cell assembly.

17 Watch the top surface of the locking nut as it is tightened. Once the locking nut's top surface is even with the cap, the shaft must be fully inserted in the sapphire bearing as directed in step 15.

The rotor will rest on top of the 90° pliers if the shaft is correctly located inside the sapphire bearing.

WARNING: DO NOT CONTINUE TIGHTENING UNTIL THIS IS VERIFIED. THE BEARINGS MAY BE DAMAGED IF THE SHAFT IS NOT INSIDE BOTH BEARINGS BEFORE TIGHTENING COMPLETELY.

AVERTISSEMENT: NE CONTINUEZ PAS À SERRER TANT CELA N'EST PAS VÉRIFIÉ. LES ROULEMENTS RISQUENT D'ÊTRE ENDOMMAGÉS SI L'ARBRE N'EST PAS À L'INTÉRIEUR DES DEUX ROULEMENTS AVANT LA FIN DU SERRAGE.

18 Replace the thumbscrew at the top of the rotary cap.

19 Turn the entire assembly right-side up and place it back into the Pressure Cell cup.

20 Insert the wrench (also called a Tommy Bar) provided into one of the six holes in the rotor assembly cap and turn the rotor assembly clockwise threading it down and into the cup.

Replacement Parts

Replacement parts for the AR Pressure Cell are available from TA Instruments. See the table below when ordering parts.

Table 4: Pressure Cell Replacement Parts

Part Number	Description
200352.002	Pressure snubber used in manifold kit
200353.001	Three-way ball valve used in manifold kit
200380.001	Wrench open-end 7/16 & 1/2
200380.002	Wrench open-end 7/16 & 9/16
200380.003	Wrench open-end 9/16 & 11/16
400.09199	Wrench (Tommy Bar) used to torque the rotor cap into the Pressure Cell cup
402815.901	Vane Rotor for DHR & AR-G2/AR 2000 Pressure Cell
402828.901	Starch Rotor for DHR & AR-G2/AR 2000 Pressure Cell
403001.901	Pressure Cell assembly
403008.901	Upper Pressure Cell assembly (with bob and magnet)
403029.901	Manifold assembly
403032.901	Alignment magnet used to hold the rotor assembly magnet in place
403040.001	Sorbothane pad used to mount the tubing manifold
403067.901	Safety pressure relief port (with 2500 psi rupture disc)
603.03519	Large Kalrez o-ring 1.296 I.D. used between rotor cap and the Pressure Cell cup
603.03557	Kalrez o-ring 0.208 I.D. used with thumbscrew on top of rotor assembly cap
613.03378	Metric hex wrench set (5, 4, 3, 2.5, 2, 1.5 mm sizes)
613.04701	90-degree angle pliers used on the rotor assembly locking nut

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