# DHR Series and AR Series

## Electrically Heated Cylinder

## Getting Started Guide

Revision B Issued June 2016

## Notice

The material contained in this manual, and in the online help for the software used to support this instrument, is believed adequate for the intended use of the instrument. If the instrument or procedures are used for purposes other than those specified herein, confirmation of their suitability must be obtained from TA Instruments. Otherwise, TA Instruments does not guarantee any results and assumes no obligation or liability. TA Instruments also reserves the right to revise this document and to make changes without notice.

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# Introduction

## Important: TA Instruments Manual Supplement

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## 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 2014/35/EU of the European Parliament and of the Council of 26 February 2014 on the harmonization of the laws of the Member States relating to the making available on the market of 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 2014/30/EU of the European Parliament and of the Council of 26 February 2014 on the harmonization of the laws of the Member States relating to electromagnetic compatibility)

EMC IEC 61326-1:2012 Ed. 2 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 instrument, as it contains no user-serviceable components.

### *Required Equipment*

While operating this instrument, 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 instrument 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.<br><br>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.

### *Cautions and Warnings*

**WARNING: Take adequate precautions prior to heating of materials if it can lead to explosion, implosion or the release of toxic or flammable gases.**

**AVERTISSEMENT: Prenez des mesures de précaution adéquates avant de chauffer des matériaux, si cela peut entraîner l'explosion, l'implosion ou le dégagement de gaz toxiques ou inflammables.**

# Table of Contents

# *Chapter 1:*

# Introducing the Electrically Heated Cylinder

## *The Electrically Heated Cylinder (EHC)*

The Electrically Heated Cylinder (EHC) is a Smart Swap™ temperature control system for concentric cylinder geometries over the temperature range ambient to 300°C. An optional cooling kit is available to improve cooling rates. It is designed for use with the Discovery Series and AR-G2, AR2000ex, and AR1500ex rheometers.

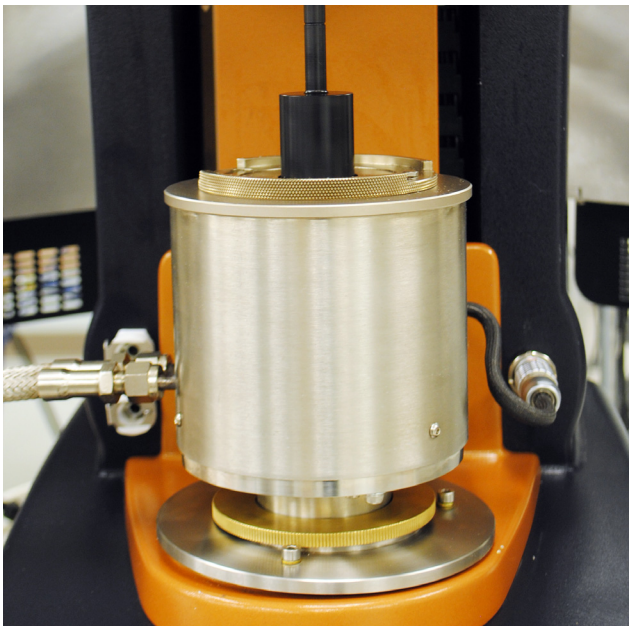

**Figure 1** Electrically Heated Cylinder.

# *About the Electrically Heated Cylinder*

The EHC (schematic shown in the figure below) uses electrical heating elements combined with air cooling to control the sample cup temperature. Heaters in the inner jacket surround the removable cup sitting inside. When cooling is required, compressed air is circulated in the cavity surrounding the jacket.

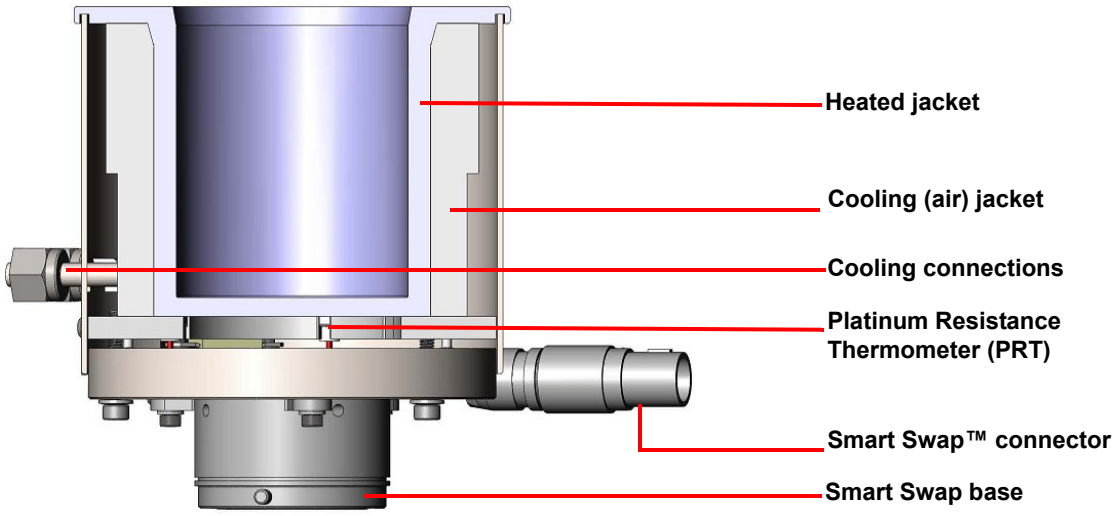


**Figure 2** Electrically Heated Cylinder system schematic diagram.

The stainless steel lower cup containing the sample fits inside the heating jacket ensuring good thermal contact. A PRT for controlling and recording the cup temperature is located in the jacket.

The air inlet/outlet connects to high temperature flexible hoses. If required, in order to prevent side loads from these stiff hoses and to avoid accidentally dislocating the jacket from the Smart Swap base, the EHC can be tightened rigidly with a locking collar to the instrument base.

## Available Rotors for the Electrically Heated Cylinder System

The following rotors are available for use with the Electrically Heated Cylinder system:

- Standard size DIN (conforms to DIN 53019)
- Recessed end
- Double gap (conforms to DIN 54453)
- Standard vane

**NOTE**: Rotors have to be rated for high temperature when the EHC is operated at temperatures above 150°C.

# *System Specifications*

Refer to the table below for Electrically Heated Cylinder system specifications:

**Table 1: Electrically Heated Cylinders System Specifications**

| | |
|---|---|
| **Temperature range** | ambient to 300°C |
| **Ramp rate** | See section below. |
| **Pt100 internal resolution** | 0.01°C |

## Ramp Rate

The maximum sustainable ramp rate will depend on a number of factors such as the start and end temperature as well as the temperature/pressure of the circulating air. To determine the maximum sustainable heating/cooling rate, perform the following test and analysis:

**1** Equilibrate to start temperature.

Perform a time sweep or peak hold test with the temperature set (if possible) to a few degrees in excess of the end temperature. Set the time much longer than you expect; the test can be aborted when the temperature has reached a stable value.

**2** Plot a graph of temperature vs. time (min) and take the derivative.

Inspect the derivative curve over your temperature range of interest. The maximum sustainable rate in °C/min will be the lowest value on the derivative curve.

# *EHC Installation Kit*

The complete Electrically Heated Cylinder system (EHC) installation kit consists of two parts:

**1** The EHC jacket kit

**2** The optional Cooling Control Unit and high temperature hoses

The EHC jacket kit includes:

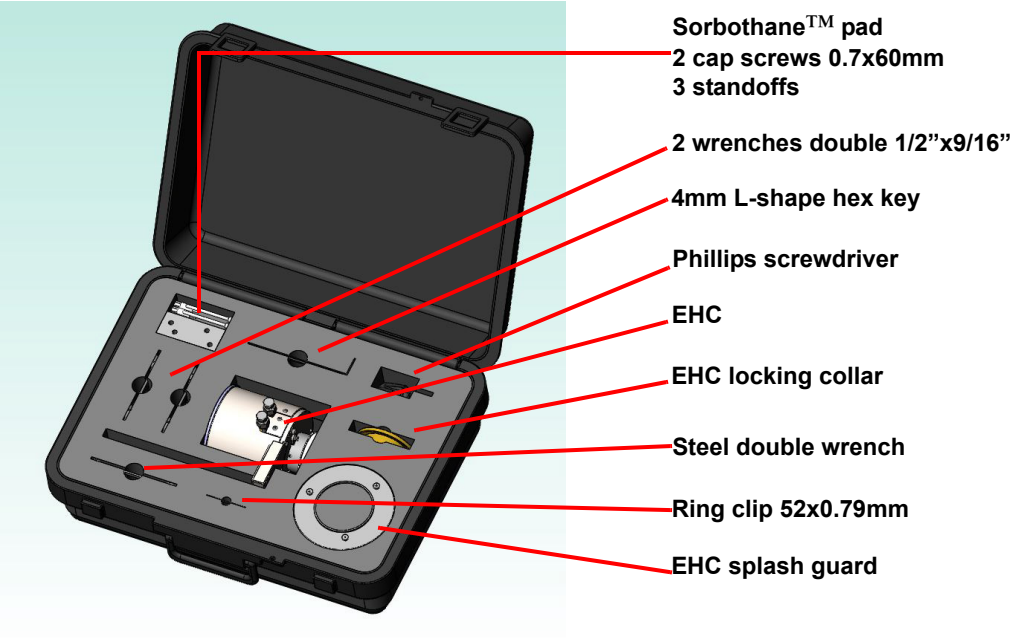


**Figure 3** EHC jacket kit.

The Cooling Control Unit kit includes:

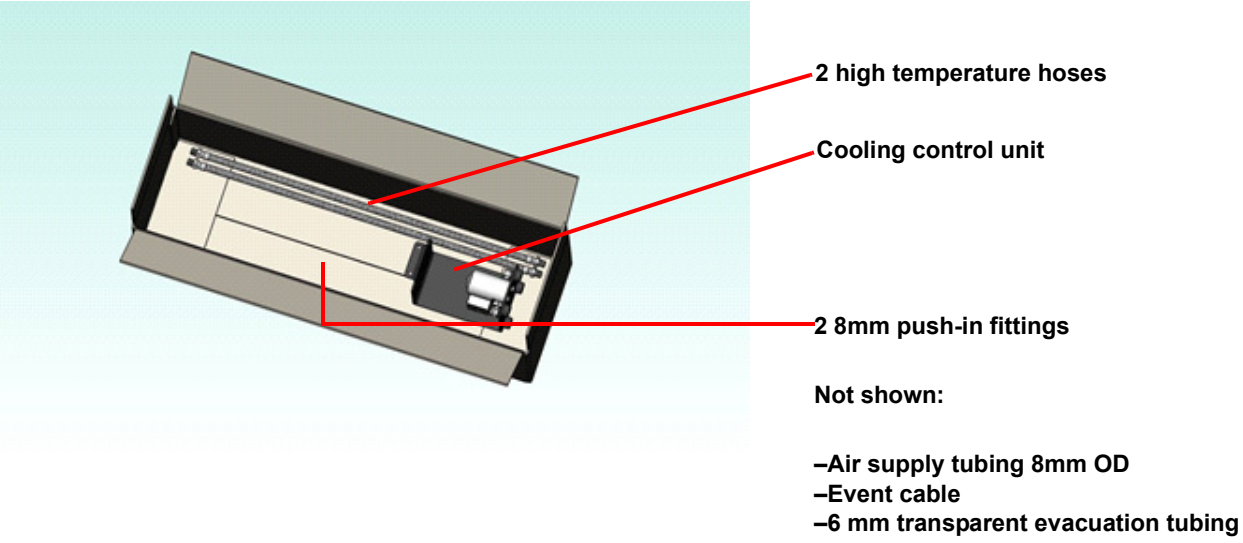


**Figure 4** Cooling Control Unit installation kit.

# *Chapter 2:*

# Installing the Electrically Heated Cylinder

The Electrically Heated Cylinder system installs on the Rheometer using a Smart Swap™ connection. Refer to the sections below for installation instructions.

## *Setting Up the Electrically Heated Cylinder*

The EHC system consists of a temperature-controlled jacket, an inner cylinder (the cup) and a rotor (or bob).

To set up the EHC on a DHR follow all of the steps below. For AR-G2/2000ex/1500ex, steps 3 and 4 are omitted.

**1** Raise the rheometer head to the top most position. If the locking collar is not going to be used, skip to step 10.

**2** Rotate the protection cover at the Smart Swap base in a counterclockwise direction and remove it.

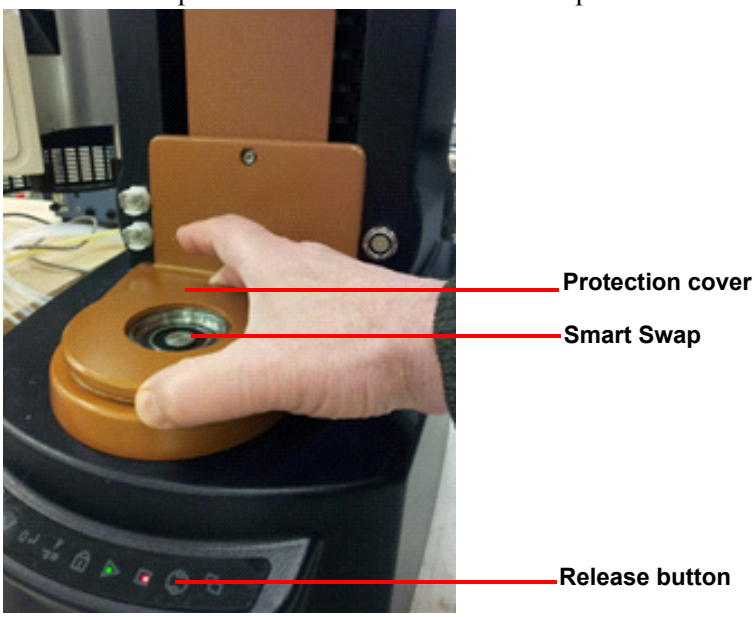


**Figure 5** Removing protection cover.

**3** Using the 4 mm hex key, remove the 3 standoff studs (see installation kit) from the base of the instrument.

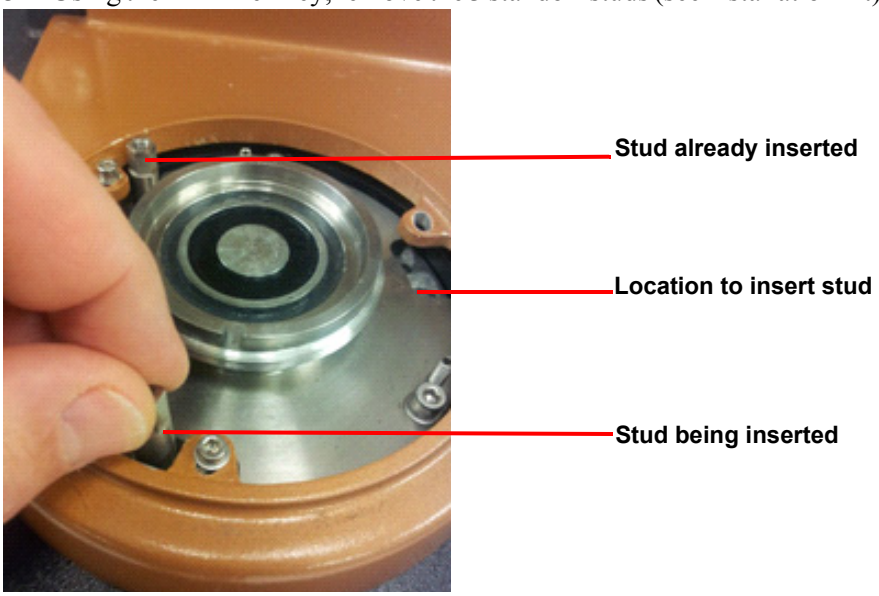


**Figure 6** Install the 3 studs to hold the protection cover.

**4** Attach the new stainless steel protection cover to the 3 standoffs at the instrument base with the captive screws.

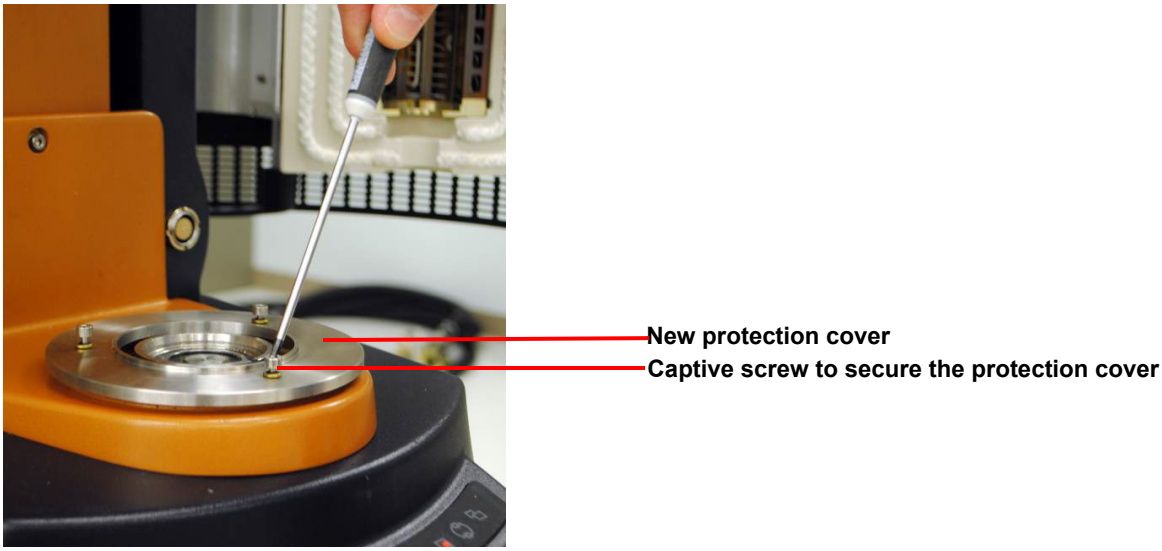


**Figure 7** Install the new protection cover.

**5** Invert the EHC.

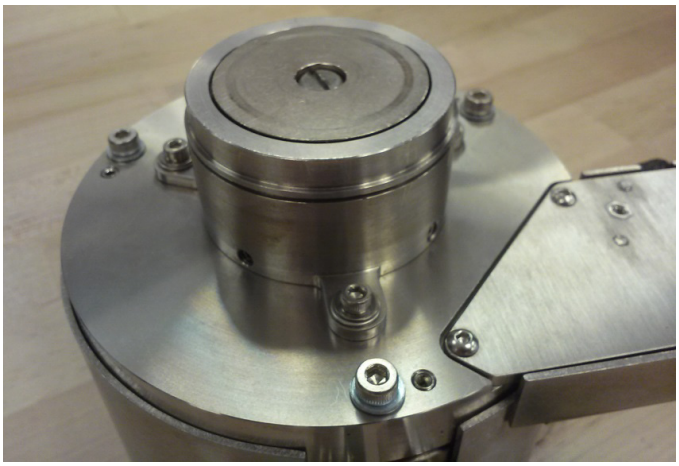

**Figure 8** Invert the EHC.

**6** Place the EHC locking collar over the Smart Swap™ coupling.

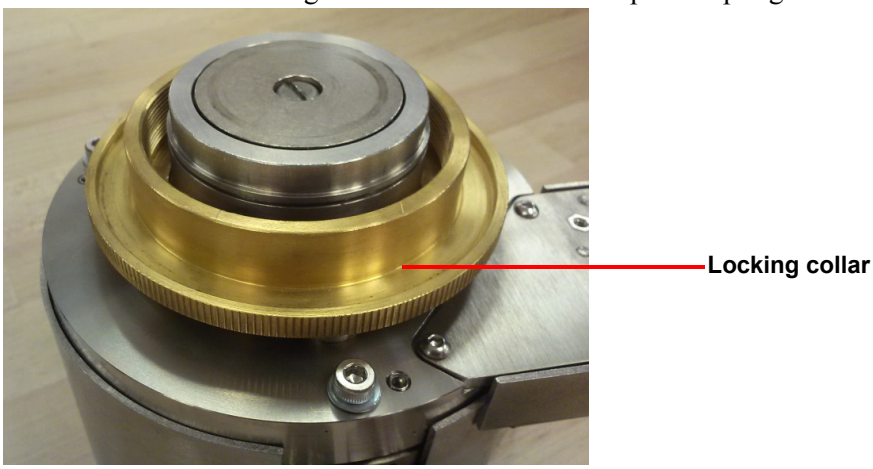


**Figure 9** Locking collar.

**7** Slide the Ring Clip over the Smart Swap.

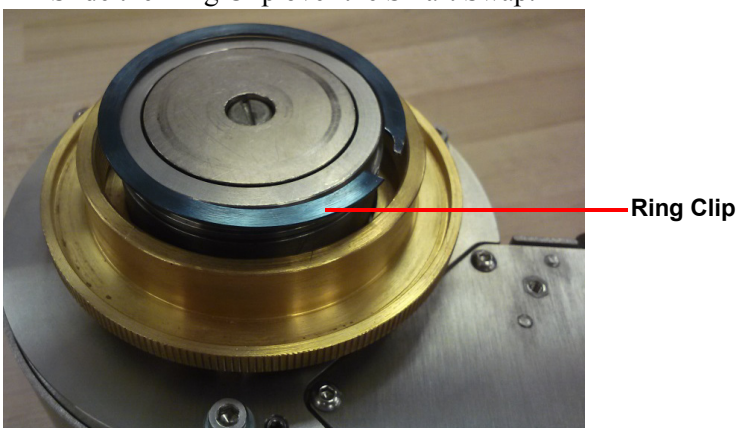


**Figure 10** Ring Clip.

**8** Start the Ring Clip on the groove by pushing one end so that it engages with the groove. Then push the rest of the clip into the groove so it is engaged all around the diameter.

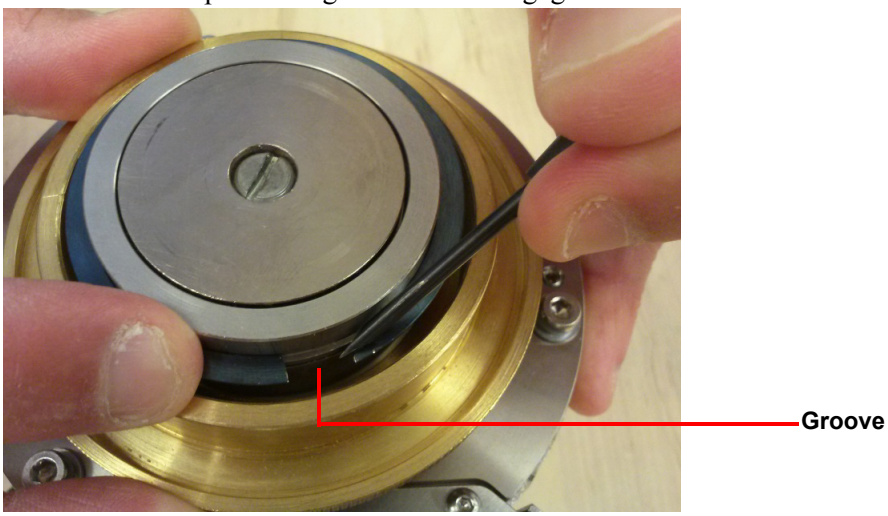


**Figure 11**

**9** Once the Ring Clip is engaged (as shown below), then the EHC can be mounted to the DHR.

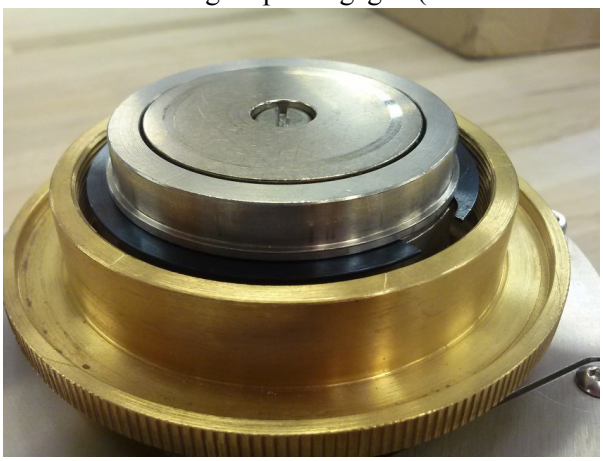

**Figure 12** Ring Clip engaged.

**10** Press the **Release** button  on the Control panel. A continuous green light indicates that the attachment can be fitted.

**NOTE**: The release state will only stay active for 10 seconds.

**11** Fit the cylinder jacket, ensuring that the cooling connectors are on the left and the alignment line faces forward. Tighten the locking collar at the Smart Swap™ base. If you are not using the locking collar, skip to step 12.

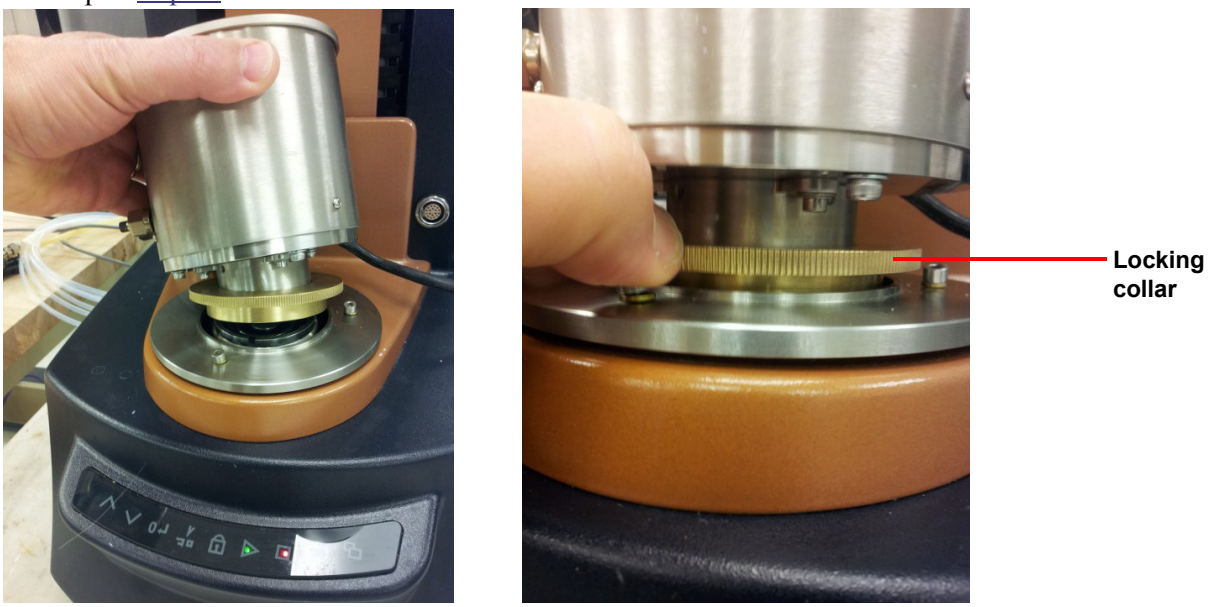


**Figure 13** Fitting the cylinder jacket (left) and tightening the locking collar at the Smart Swap base (right).

**12** When the green status light turns off, the lower cup is correctly installed.

**13** Connect the power cable.

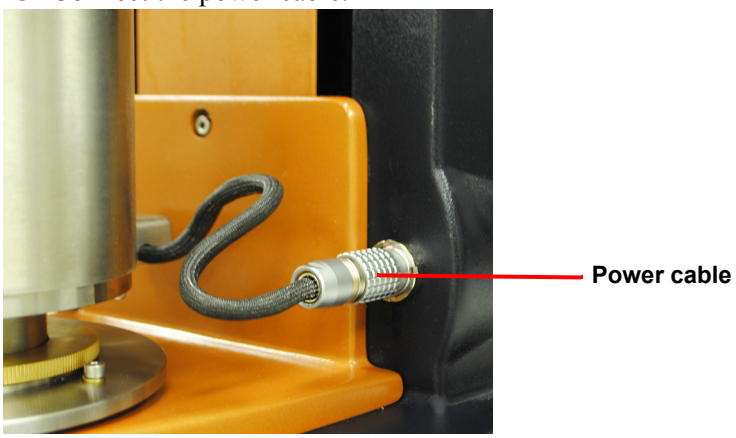


**Figure 14** Connecting the power cable.

## Installing the Optional Cooling Control Unit

Skip to step 18 if you do not have the Cooling Control Unit.

**14** Using the two supplied wrenches, remove the plugs from the cooling connectors on the EHC.

**15** Remove the caps from the end of the two high temperature-reinforced hoses. Attach and tighten one end of the reinforced hoses to the cooling connectors.

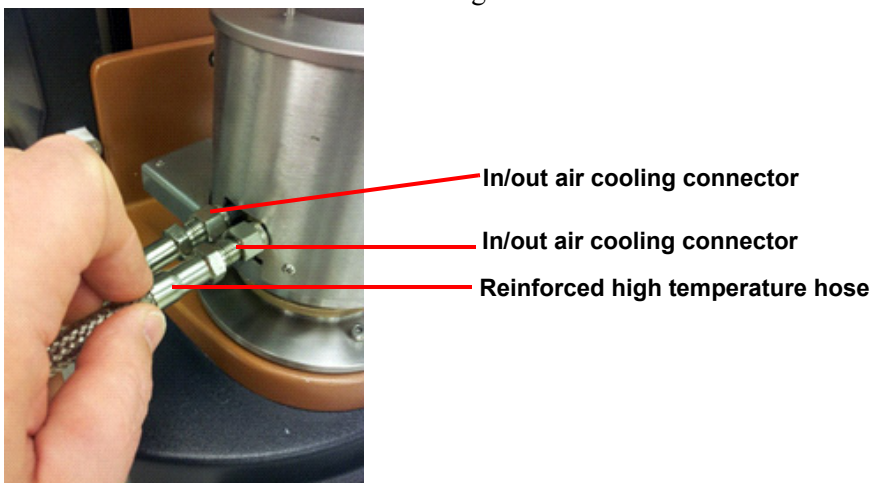


**Figure 15** Connecting the reinforced hoses to the EHC.

**16** Connect one hose to **Liquid Out** port on the Cooling Control Unit outlet. Connect and tighten the transparent evacuation tubing to the other reinforced hose.

**WARNING: At the start of the cooling cycle, the 6 mm high temperature reinforced hoses and evacuation tubing will become hot due to the EHC purging. These hoses/tubing should not come in contact with other cabling or hoses. Direct the open end of the evacuation hose away from people to avoid any harm. It is recommended to evacuate the cooling air into a hood or directly outside of the building. Make sure the end of the evacuation hose is securely fastened to prevent any harm.**

**AVERTISSEMENT: Au début du cycle de refroidissement, la température élevée 6mm renforcé tuyaux et de tubes d'évacuation devient chaud en raison de la purge EHC. Ces tuyaux / tubes ne doit pas entrer en contact avec d'autres câbles ou tuyaux. Diriger l'extrémité ouverte de l'évacuation flexible loin des gens pour éviter tout dommage. Il est recommandé d'évacuer l'air de refroidissement dans une hotte ou directement à l'extérieur du bâtiment. Assurez-vous que l'extrémité du tuyau d'évacuation est solidement fixé pour éviter tout dommage.**

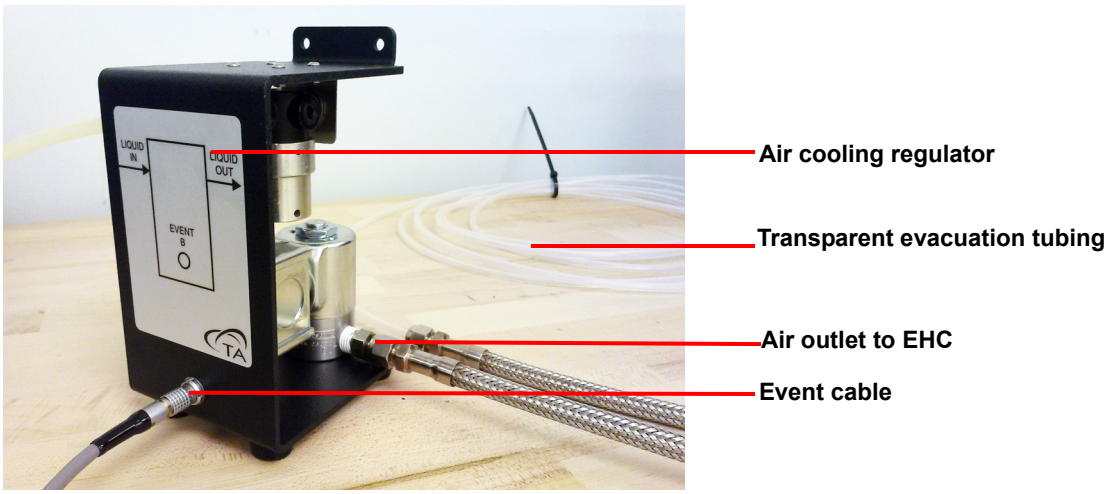


**Figure 16** Connecting the reinforced heat sink hoses and the event cable.

**17** Use the 8 mm tubing and Y-piece to break into the rheometer air supply before the filter regulator, and connect to the **Liquid In** port on the Cooling Control Unit inlet.

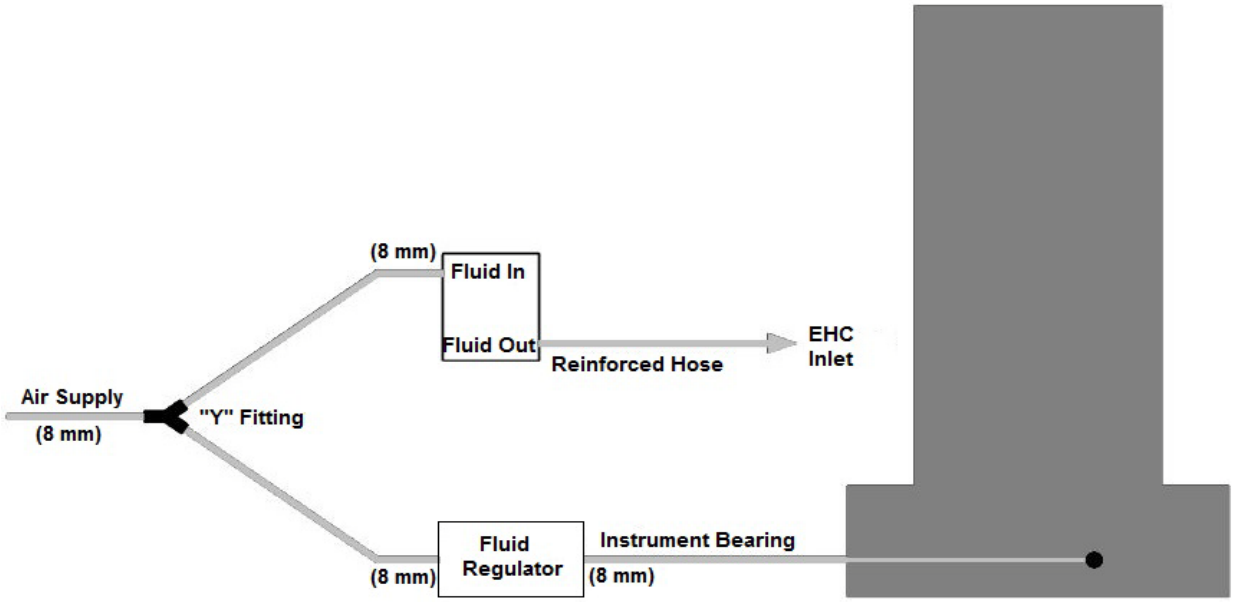


**Figure 17** Air supply connections.

**18** Connect the Event cable to the **Event B** port on the regulator and the **Event Port B** at the rear of the rheometer.

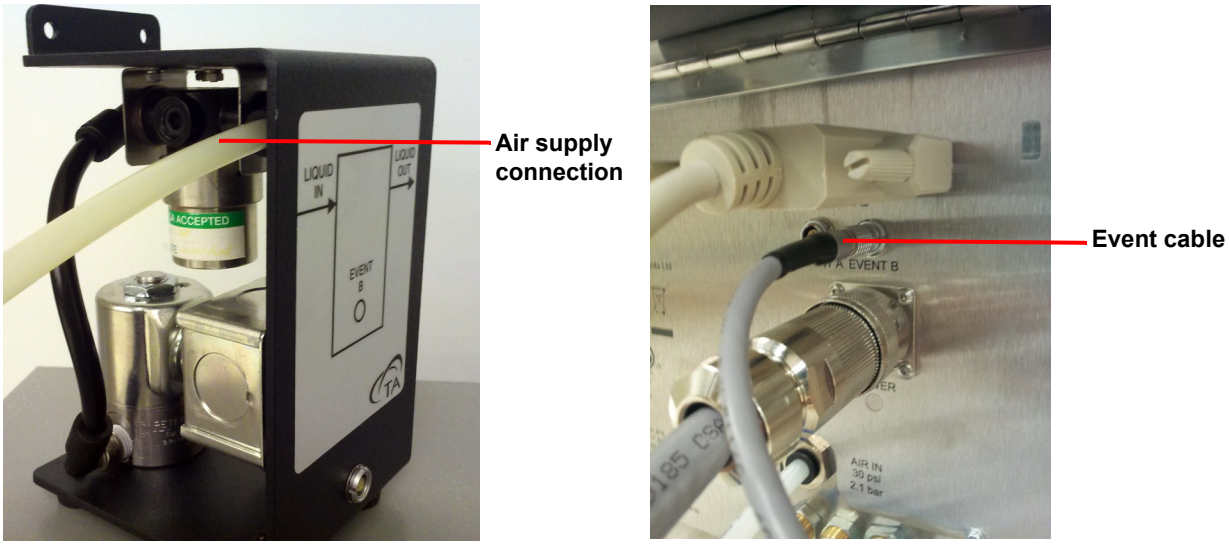


**Figure 18** Connecting the air supply to the Cooling Control Unit (left); Connecting the Event cable to the rear of the rheometer (right).

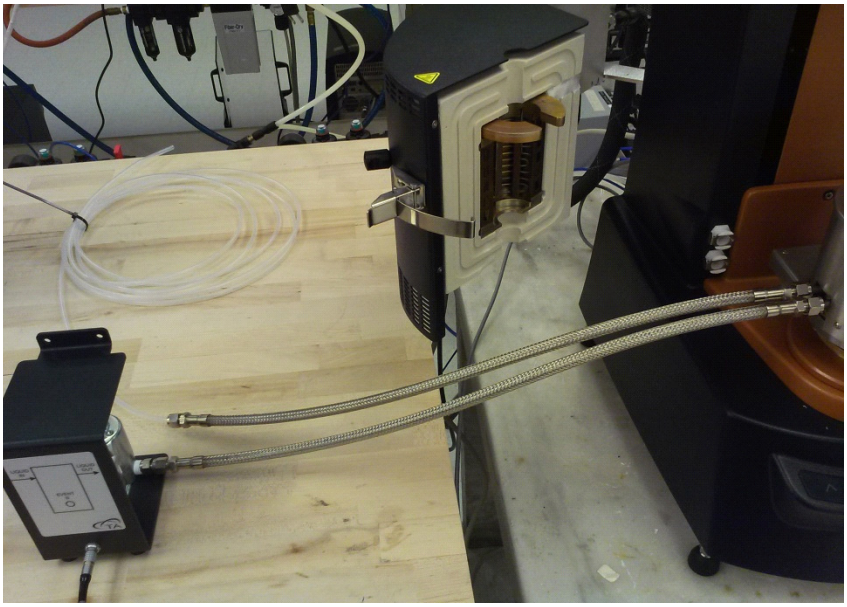

**Figure 19** Typical hose arrangement.

Next, see "Installing the Lower Cup Geometry" on page 20 and "Attaching the Rotor and Finding the Zero Position" on page 21.

## Removing the EHC

1 Press the **Release** button on the Control panel. A flashing green light indicates that the cylinder jacket can now be removed.

2 Press the **Release** button again. A continuous green light indicates that you can remove the cylinder jacket.

3 Remove the EHC from the rheometer. Remove the stainless steel protection cover (DHR series only) and screw on the gold-colored cover by rotating it clockwise. Remove the event cable at the rear of the test station.

# *Installing the Lower Cup Geometry*

Before installing the rotor, the lower cup must first be installed.

1 Loosen the two locking screws on the top of the EHC.

2 Before inserting the cup, make sure that the surfaces of the cup and jacket are clean. Gently place the cup into the jacket and twist into place. Tighten the two locking screws by hand.

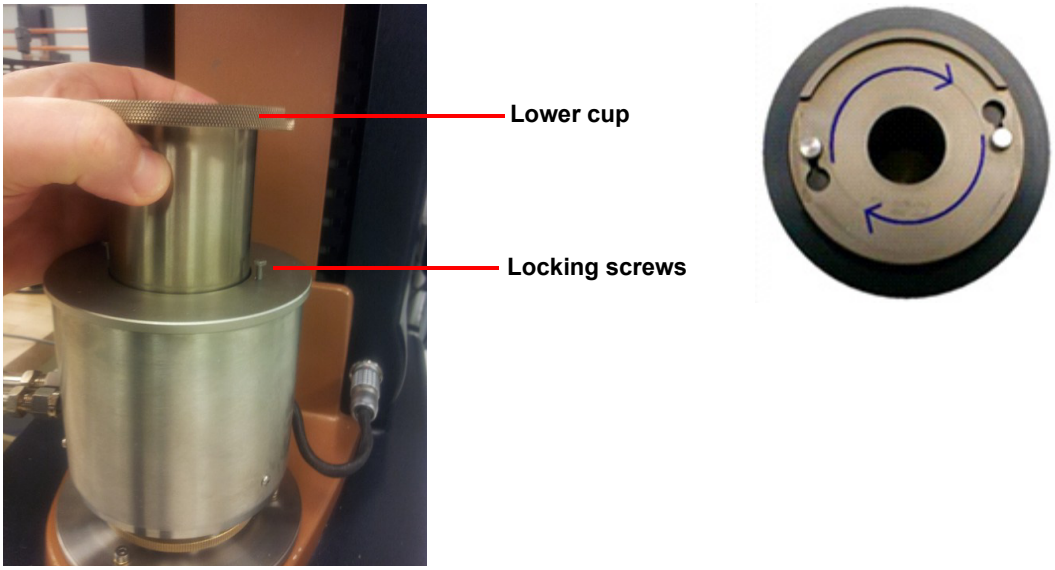


**Figure 20** Installing the lower cup (left); Rotate cup clockwise to clamp and counterclockwise to release (right).

**NOTE**: The fit between the cup and the jacket is tight by design for best thermal conductivity. Take care not to force the cup into place. The cup will be easier to fit if the jacket is heated to 10–20°C above ambient.

# *Attaching the Rotor and Finding the Zero Position*

**NOTE**: Rotors have to be rated for high temperature when the EHC is operated at temperatures above 150°C.

Finding the zero gap position for the rotor is slightly more difficult than for the geometries used with other temperature systems, as the gap between the rotor and the bottom of the cup cannot be observed visually. It is a good idea to place the bob in the cup before attaching it to the draw rod, to ascertain the approximate zero position. Look for the position of the rotor shaft relative to the top of the cup. On most geometries, a datum mark is machined on the shaft to help with finding the zero position.

**1** Attach the rotor to the draw rod.

**2** To find the zero position, lower the instrument head until the datum mark on the shaft of the rotor is level with the top of the cup.

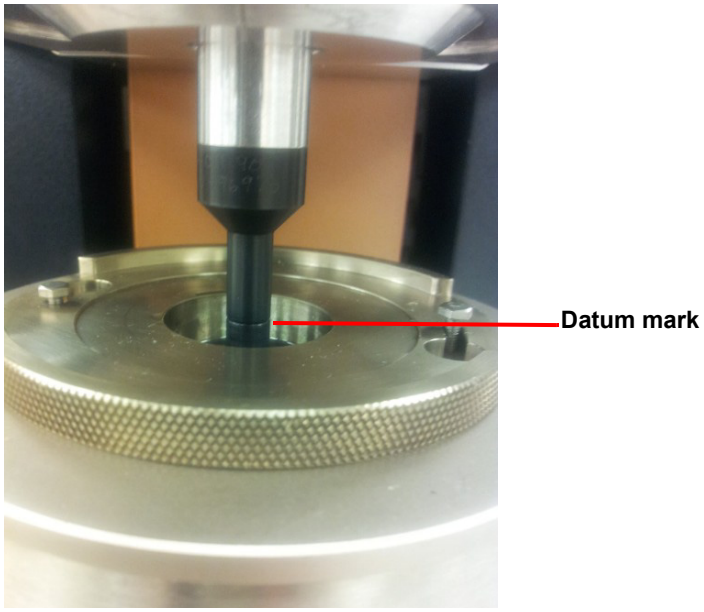


**Figure 21** Finding zero position using the datum mark (DHR Series shown).

**WARNING: Operating at high temperature can be hazardous. Use appropriate protection such as gloves and goggles.**

**AVERTISSEMENT: L'utilisation à température élevée peut être dangereuse. Utiliser une protection appropriée, comme des gants et des lunettes.**

You can now set up the measuring geometry in the rheometer software and set the gap as explained in TRIOS Online Help.

# *Changing the Cup*

To change the size of the cup:

**1** Undo the two locking screws on the cup. Turn and lift out the cup as shown in the Figure 20.

**2** Before inserting the replacement cup, make sure that the surfaces of the cup and jacket are clean. Gently place the cup into the jacket and twist into place. Tighten the two screws by hand.

**NOTE**: The fit between the cup and the jacket is tight by design for best thermal conductivity. Take care not to force the cup into place. The cup will be easier to fit if the jacket is heated to 10–20°C above ambient temperature before inserting.

# *Pressure Cell*

The Pressure Cell can be used in conjunction with the EHC up to 300°C.

**Figure 22** Pressure cell mounted in the EHC jacket.

**NOTE**: When using the Pressure Cell with the EHC, add an additional Sorbothane™ insulation block between the manifold mounting plate and the manifold.

To add the additional Sorbothane insulation block:

**1** Remove the manifold from the mounting plate by unscrewing the two cap head screws at the back of the plate.

2 Insert the additional Sorbothane insulation pad. Reinsert the mounting plate and tighten with the longer cap screws, supplied with the EHC kit.

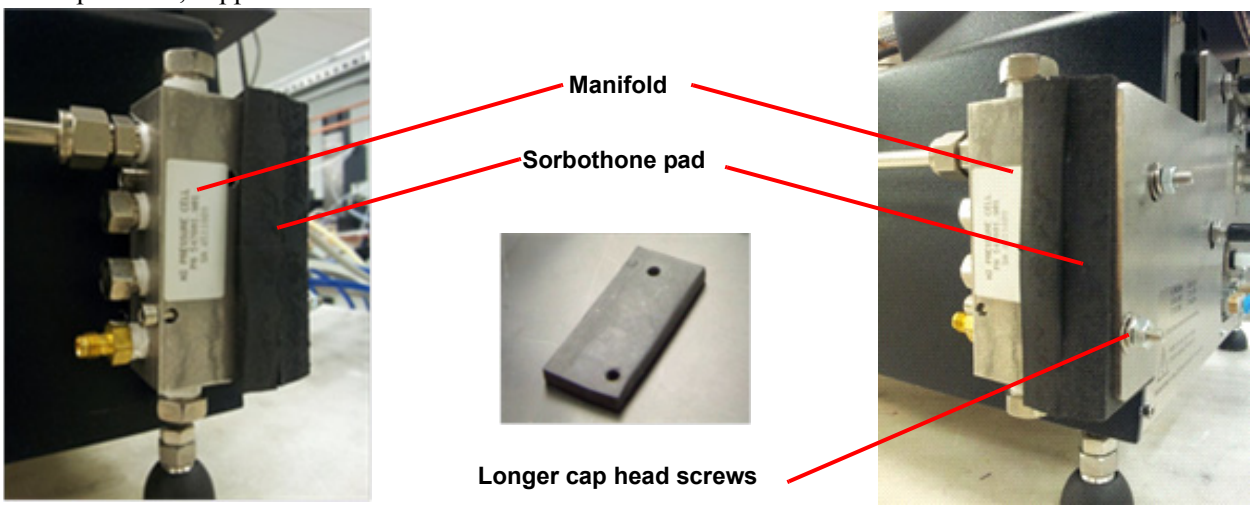


**Figure 23** Modify the Pressure Cell manifold plate.

## *Concentric Cylinder Solvent Trap Cover*

The optional solvent trap for the Peltier Concentric Cylinder can be used with the EHC. It includes a base reservoir and a two-piece cover that is mounted to the shaft of the rotor. The solvent trap provides a vapor barrier to seal the environment inside the cup and prevents solvent evaporation.

**NOTE**: Make sure that the solvent trap fluid has a boiling point above testing temperature.

**Figure 24** Concentric Cylinder Solvent Trap.