DHR Series and AR Series

Electrically Heated Plate

Getting Started Guide

Revision E Issued November 2013

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

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 instrument, 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 Electrically Heated Plates

About the Electrically Heated Plates

The Electrically Heated Plates (EHP) consist of a high temperature parallel plate and a cone and plate Smart Swap™ temperature system. It designed for use with the Discovery Series and AR-G2, AR2000ex, and AR 1500ex rheometers. The EHP is commonly used for the testing of polymer melts. On the Discovery Series and AR-G2, the temperature of the upper plate is measured directly using the Upper Temperature Sensor (UTS). This consists of a special drawrod containing a Pt100 temperature sensor, the information from which is transmitted to a remote reading device.

The EHP is cooled by air passing through ducts. The air flow is controlled manually. The EHP includes a heated purge. Operation can be either in air or under nitrogen or other inert gas. A gas purge cover is provided for operation under inert gas.

An optional low temperature cooling accessory, the Gas Cooling Accessory (GCA), is also available for temperature control down to –70°C.

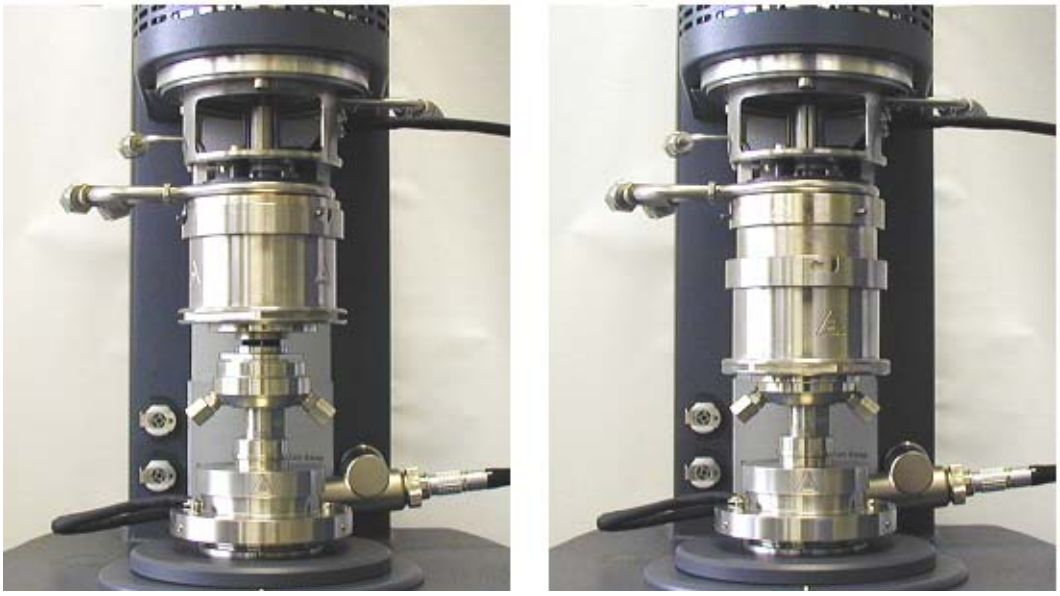

Figure 1 The EHP with cover up on left, and cover down on right (AR Series shown).

Electrically Heated Plates Components

The EHP consists of three main components (see the figure below).

- An upper fixture that attaches to the rheometer head. This fixture contains electrical heating elements, cooling ducts, and purge duct.
- A lower fixture that mounts on the rheometer Smart Swap connector. This fixture contains electrical heating elements and a cooling duct.
- A geometry holder that attaches to the rheometer rotating shaft. The geometry holder contains a heat spreader. There is no physical contact between this component and the upper fixture. For the Discovery Series and AR-G2, the geometry holder is supplied as a single unit, with a fixed geometry. For the AR2000ex and AR 1500ex, the geometry can be removed from the holder.

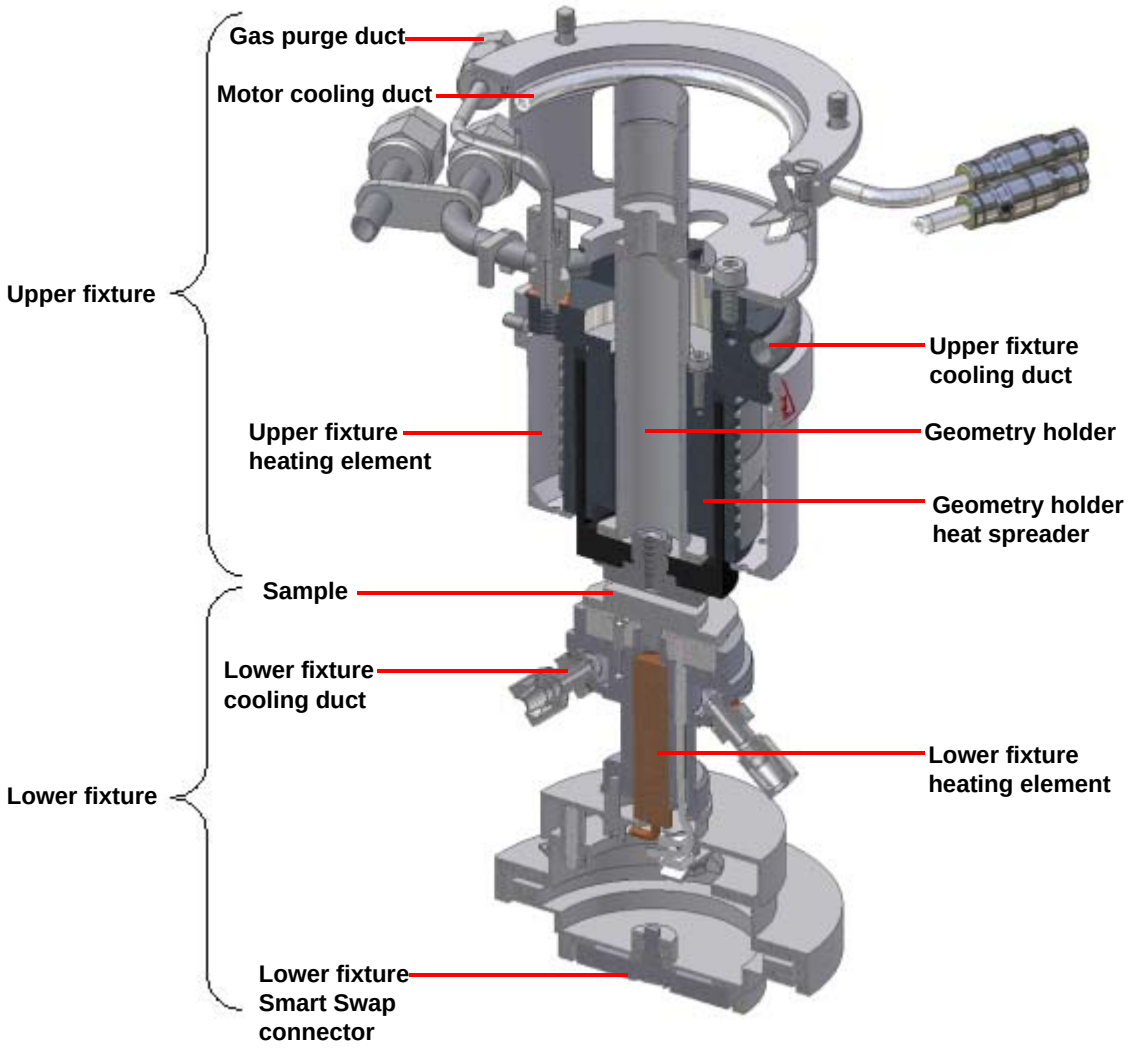

Figure 2 EHP components.

In addition to the three main components, a supplied pneumatic manifold containing flow meters mounts on the rear of the rheometer. The 6-mm outer diameter high-temperature tubing required to connect this unit to the cooling ducts of the upper and lower fixtures is supplied cut to length and with the appropriate connectors attached.

WARNING: Do not use any tubing other than that provided by TA Instruments to connect the bracket to the upper and lower fixtures. If replacement tubing is required, please contact your local TA Instruments' representative or service engineer.

AVERTISSEMENT: N'utilisez pas une tuyauterie autre que celle fournie par TA Instruments pour raccorder le support aux fixations supérieure et inférieure. S'il est nécessaire de remplacer la tuyauterie, contactez votre représentant local de TA Instruments ou l'ingénieur d'entretien.

Heating of the EHP is through the electrical elements in the upper and lower fixtures. Cooling is provided by air carried in the coolant ducts. The air flow rate is manually controlled.

The temperature of the lower fixture is read by a Pt100 probe placed close to the surface of the lower plate, and in close thermal contact with it.

System Specifications

Refer to the table below for EHP system specifications:

Table 1: Electrically Heated Plate System Specifications

Temperature range	Ambient to 400°C
Maximum heating rate	30°C/min (uncontrolled) 10°C/min (controlled) ramp rate*
Crash cooling flow rate	No limits.
Purge gas flow rates	5 L/min recommended (Set the lowest flow below 5 L/min that stops oxidation. This will reduce noise and residuals during sensitive creep tests.)

* Ramp rate: The maximum sustainable ramp rate will depend on a number of factors such as the start and end temperature and the temperature / flow rate of circulation fluid. 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 will be the lowest value on the derivative curve.

Chapter 2:

Installing the Electrically Heated Plates

The steps needed to attach the EHP to the rheometer involve the following:

- Mounting and connecting the manifold.
- Mounting and connecting the upper and lower fixture.
- Attaching the geometry holder.
- Attaching the purge gas cover.

The following topics are also covered in this section:

- Removing the geometry holder.
- Removing the purge gas cover.

Installing the EHP

Follow the instructions below.

Mounting and Connecting the Manifold

Follow these instructions to install the EHP manifold:

1 Ensure that the main air supply is turned off and depressurized.

2 Mount the manifold to the rear of the rheometer using the four crosshead screws provided. For a DHR instrument, mount the adapter bracket onto the rear of the rheometer and then mount the manifold to the adapter.

A schematic of the main air and purge gas inlet connections to the rheometer and manifold is shown in Figure 4 on the next page. Refer to the schematic for the remaining instructions.

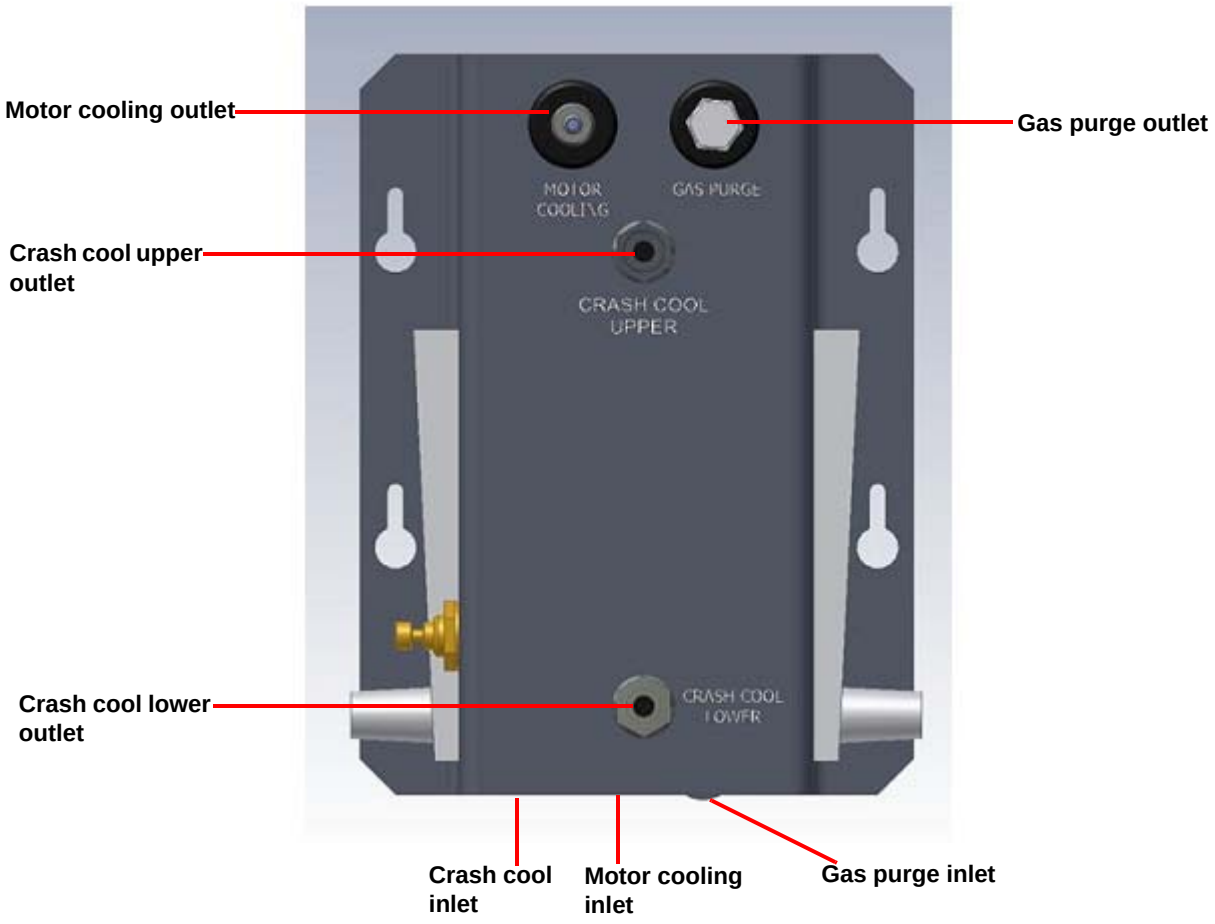

Figure 3 EHP manifold.

3 Disconnect the main air supply from the instrument filter regulator system, and attach the 8-mm ‘Y’ piece provided to the main air line.

4 Connect one branch of the ‘Y’ piece to the filter regulator system using the 8-mm outer diameter white/natural tubing provided.

5 Insert the 8-mm to 6-mm reducer into the second branch of the ‘Y’ piece. Then connect this to the 6-mm ‘Y’ piece using the 6-mm outer diameter white/natural nylon tubing.

6 Connect one branch of the 6-mm ‘Y’ piece to the crash cool inlet port on the manifold using the 6-mm outer diameter white/natural nylon tubing.

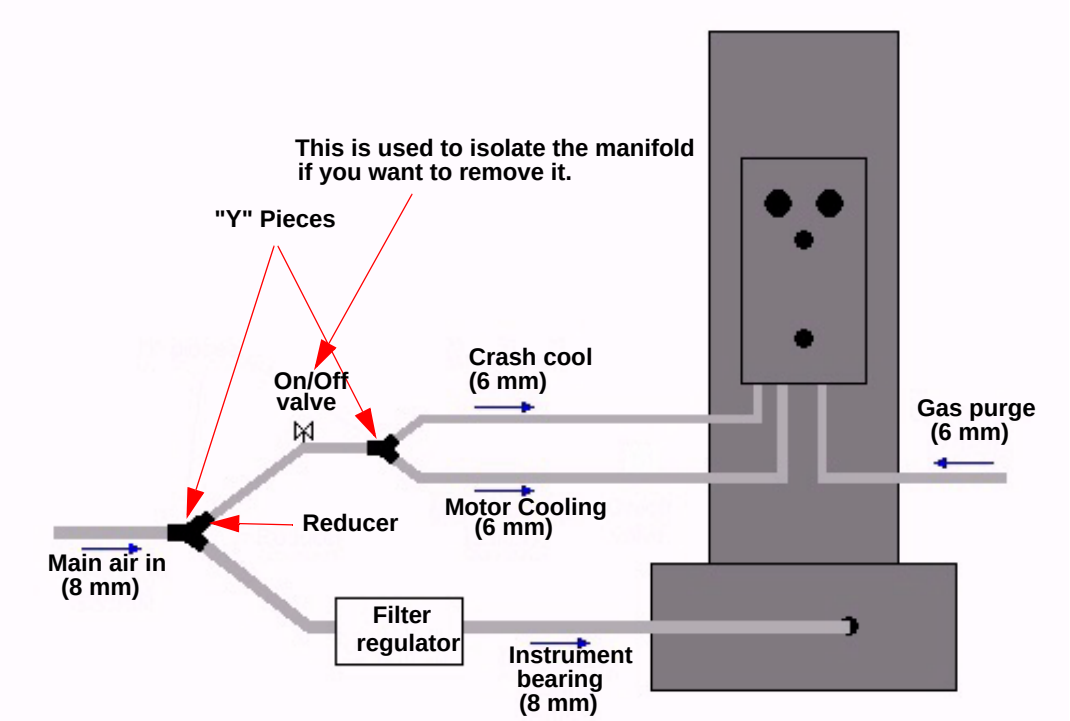

Figure 4 Schematic of air and gas purge from the manifold to the rheometer.

7 Connect the other branch of the ‘Y’ piece to the motor cooling inlet port on the manifold using the 6-mm outer diameter white/natural nylon tubing.

NOTE: The motor cooling inlet and outlet, and gas purge inlet connectors are Swagelok® fittings that you will need to install. Information on the correct procedure for this is given on the Swagelok company website at www.swagelok.com.

8 If inert gas is required, connect the gas purge supply to the gas purge inlet on the manifold using the 6- mm outer diameter white/natural tubing. The purge gas should be nitrogen or other inert gas used to prevent oxidation of the sample by contact with air. The maximum pressure for the purge gas is 100 psi (6.9 bar).

Mounting and Connecting the Upper and Lower Fixtures

Follow the instructions below to install the upper and lower fixtures on the rheometer. Refer to the figure below, which shows the upper and lower fixtures mounted, for reference.

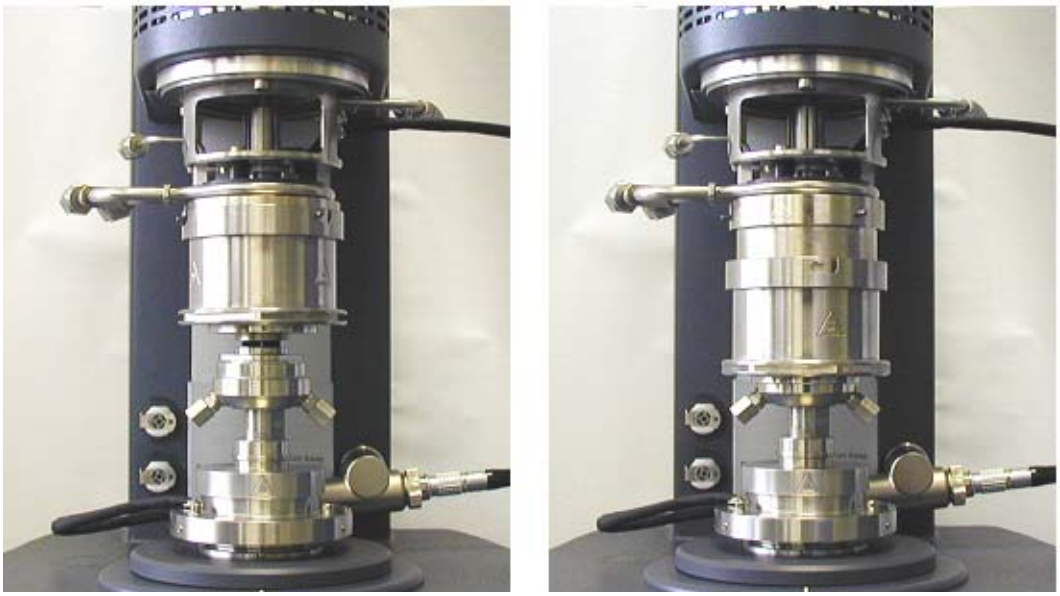

Figure 5 The EHP with cover up on left, and cover down on right (AR Series shown).

1. Ensure that the rheometer is turned on. Raise the head to the maximum (use the **Head UP** button located on the instrument control panel).
2. Attach the upper fixture to the mounting ring on the underside of the instrument head using the three captive screws provided. Note that the power cable should project to the right of the instrument when viewed from the front.
3. Mount the lower fixture on the rheometer using the Smart Swap connection.
4. Connect the upper and lower fixture cables to the right and left sockets respectively on the Smart Swap EHP adapter. For proper system identification, it is important that this step be completed before step 5.
5. Connect the Smart Swap EHP adapter to the Smart Swap socket (see Figure 6).

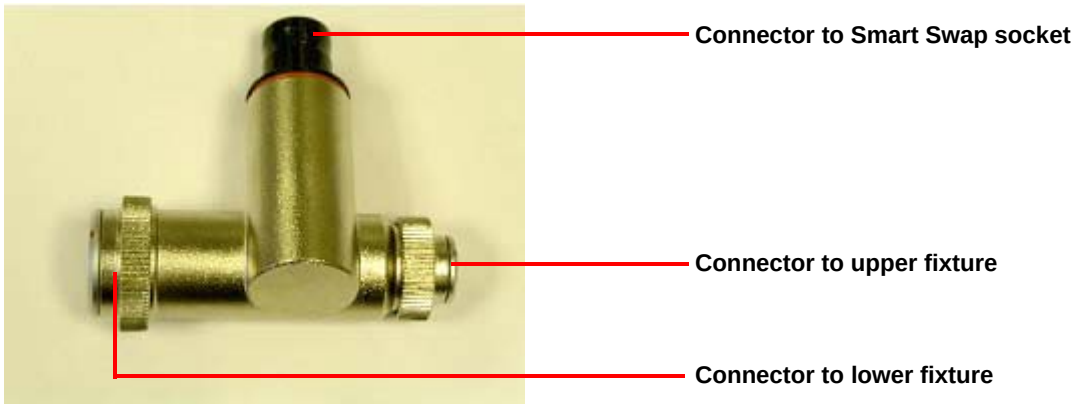

Figure 6 Smart Swap adapter connections.

A schematic of the cooling air and gas purge outlet connections from the manifold is shown in the figure below.

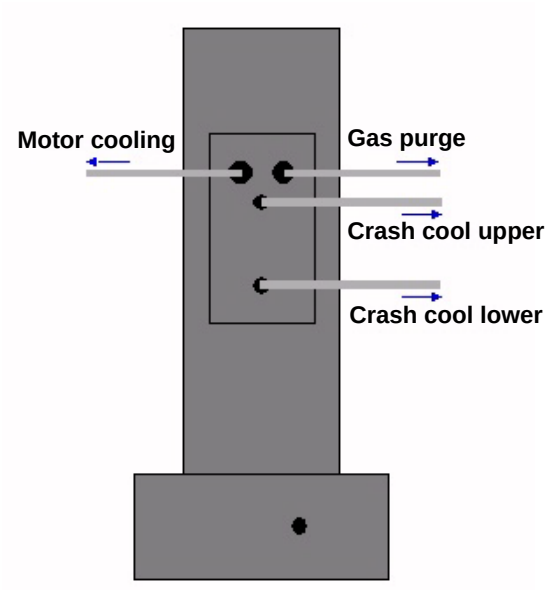

Figure 7 Schematic of cooling air and gas purge outlets from upper and lower fixtures.

6 Remove the plugs from the connectors on the cooling ducts.

7 Connect the motor cooling and crash cool upper and gas purge outlets on the manifold to the appropriate inlets on the upper fixture using the fittings provided. See the figure below.

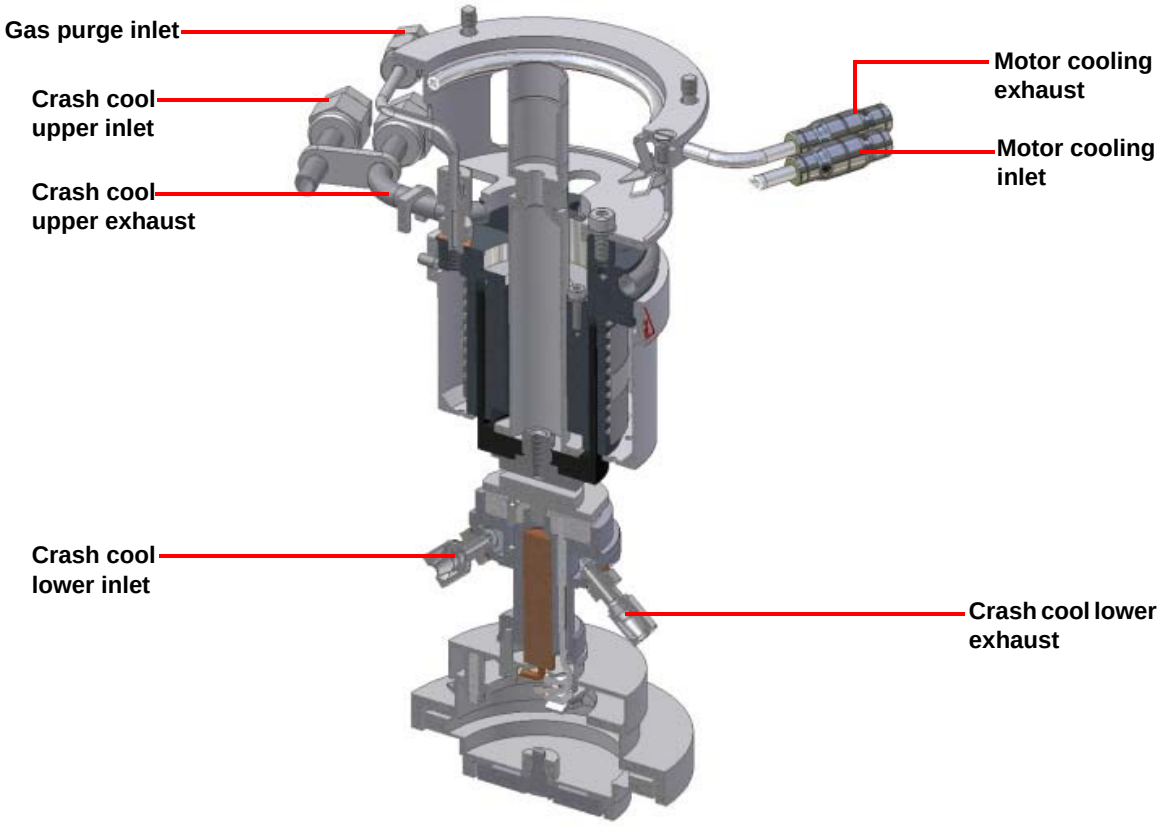

Figure 8 Cooling air and purge gas connections to the upper and lower fixtures.

8 Connect the crash cool upper exhaust pipe and lower exhaust pipe to the crash cool upper and lower outlets, respectively, using the compression fittings provided. Ensure that the pipes are pointing away from the flexible tubing.

Figure 9 Crash cool upper exhaust pipe.

Attaching and Removing the Geometry Holder

The geometry holder and geometry holder spanner are shown in the figure below. The holder used with the Discovery Series and AR-G2 is a single piece, with fixed geometry. The holder used with the AR 2000ex and AR 1500ex has a removable geometry.

Figure 10 Geometry holder (left) and geometry holder spanner (right).

Attaching the Geometry Holder

Follow the instructions below to install the geometry holder onto the rheometer:

1 Ensure that the rheometer is turned on. Raise the head to the maximum (use the **Head UP** button located on the instrument control panel).

2 *For the AR2000ex and AR 1500ex only*: Attach the geometry to the holder and tighten using the geometry holder spanner.

3 Carefully insert and position the holder within the upper fixture, and connect it to the instrument shaft by rotating the drawrod.

4 Tighten the geometry holder by grasping it on the machined flats at the base of the heat spreader with the geometry holder spanner, and rotating the drawrod.

Removing the Geometry Holder

Follow the instructions below to remove the geometry holder:

1 Ensure that the rheometer is turned on. Raise the head to the maximum (use the **Head UP** button located on the instrument control panel).

2 Grasp the geometry holder on the machined flats at the base, and unscrew from the instrument shaft by rotating the drawrod.

3 Lower the geometry holder carefully until it is clear of the upper fixture, and remove from the rheometer.

Attaching and Removing the Purge Gas Cover

The purge gas cover fixing tool and cover are shown in the figure below. The fixing tool should always be used to handle the hot cover.

Figure 11 The purge gas cover fixing tool (left) and purge gas cover (right).

Attaching the Purge Gas Cover

Follow the instructions below to install the purge gas cover:

1 Raise the instrument head (use the **Head UP** button located on the instrument control panel).

2 Use the tool to lift the cover by engaging with the slots on the lover cover rim, as shown in the figure below.

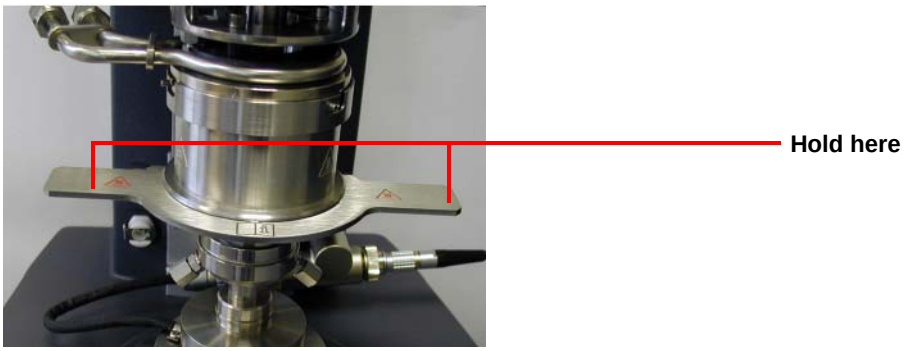

Figure 12 Using the purge gas cover fixing tool (AR Series shown).

CAUTION: Gloves should be worn when handling the tool, as it can get hot.

MISE EN GARDE: Portez des gants lors de la manipulation de l'outil, car il peut devenir chaud.

3 Slide the cover over the upper fixture, engaging the bayonet fittings on the cover with the lugs on the upper fixture.

4 Twist the cover to hold in place and remove the fixing tool. The cover is now in the up position, as shown in the figure below.

Figure 13 Purge gas cover in up position (AR Series shown).

NOTE: The fitting tool may not fit if the temperature difference between the cover and the tool is too large. In that case, cool the UHP before attempting to remove the cover.

Running Experiments Using the Purge Gas Cover

While an experimental procedure is running, the cover should be in the DOWN position, as shown in the figure below.

Figure 14 Purge gas cover in down position (AR Series shown).

To lower the purge gas cover, twist the fitting tool slightly to disengage from the lugs on the upper fixture, and lower the cover. The cover should sit squarely on the rim on the lower fixture.

Remove the fixing tool after lowering the cover.

Removing the Purge Gas Cover

Follow these instructions to remove the purge gas cover:

1 Raise the instrument head (use the **Head UP** button located on the instrument control panel).

2 Lower the cover using the fitting tool, and bring it clear of the instrument upper and lower fixtures.

About the View Cover

An optional extra for the EHP is the view cover, shown below. This is designed to allow the sample to be seen by the user, and replaces the purge gas cover. The view cover can also be used with nitrogen or other inert gas. To attach and remove the view cover, use the same procedure as that used for purge gas cover.

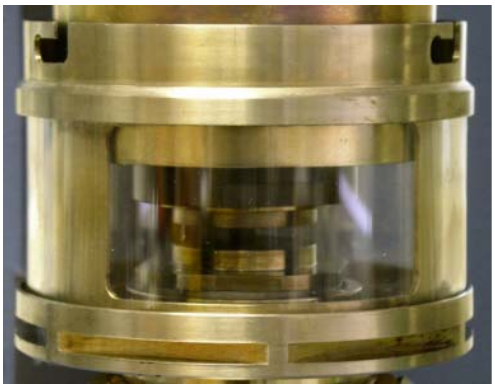

Figure 15 The view cover shown in place on the EHP.

NOTE: The view cover should not be used above 300°C.

Setting the Gas Flow Rates

The EHP cooling and purge gas flow rates are operated manually. The regulators and flow meters are mounted on the sides of the manifold. The gas purge regulator and flow meter are on the left of the manifold, the motor cooling regulator, and flow meter; the crash cool regulator is mounted on the right of the manifold, when viewed from the front.

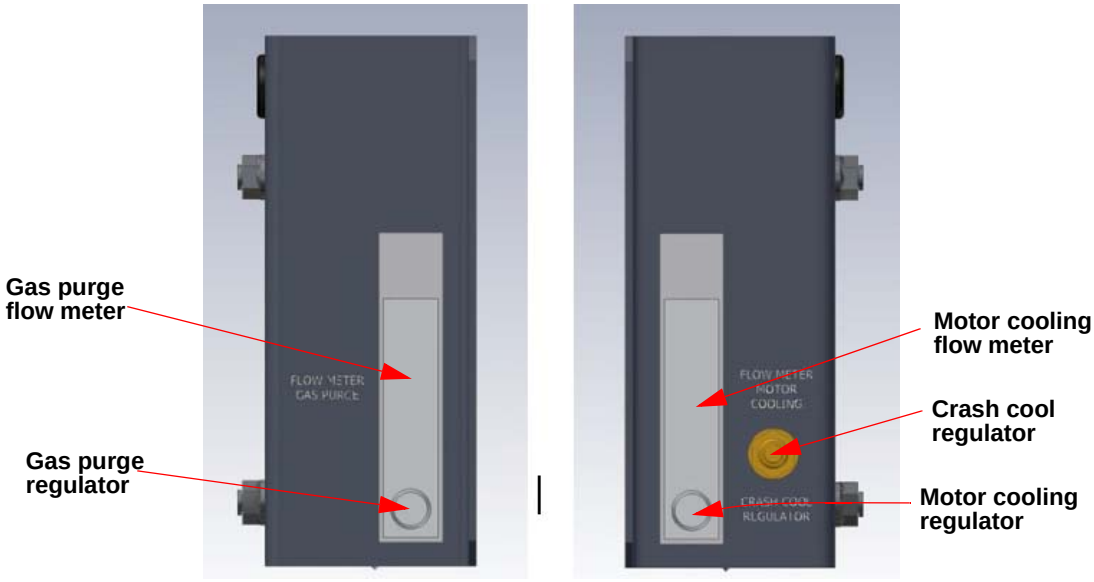

Figure 16 Manifold left and right side views.

Follow these guidelines when setting the gas flow rates:

- If required, use the purge gas regulator to set the purge gas flow rate to a maximum of 5 liters per minute (Lpm).
- It is only necessary to use the motor cooling when the temperature of the EHP is greater than 250°C, in which case the gas flow rate should be set to 10 Lpm.
- Use the crash cool regulator if rapid cooling of the EHP is required.

NOTE: When using the EHP, avoid contaminating the mating surfaces of the cover and upper and lower fixtures with sample, since this may impede removal of the cover.

EHP Temperature Calibration Factors

Platinum Resistance Thermometers (PRT's) are located in the lower pedestal, upper jacket assembly, and Upper Temperature Sensor (UTS) (this applies to the Discovery Series and AR-G2 with standard plates only). Depending on your configuration, it may be necessary to enter calibration factors into TRIOS Software.

Discovery Series and AR-G2 with Standard Plates

The PRT's in the EHP lower and UTS are uniquely calibrated with a traceable dry block temperature system during the manufacturing process. These calibration values are stored within the component and are used automatically by the system. You will not need to enter these values.

Discovery Series and AR-G2 with Disposable Plates/AR2000/ AR2000ex/AR 1500ex

Without the UTS, the temperature of the upper plate can only be inferred by calibration, using a zero heat flow sensor that matches the calibrated PRT in the EHP lower with the PRT in the upper jacket assembly. This calibration is carried out during the manufacturing process and the calibration factors are supplied on a certificate with every EHP system. To enter these factors, follow the instructions on the certificate.

NOTE: If you also have a Discovery Series or AR-G2 with standard plates, the system will automatically configure itself when you change between the two configurations.

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