DHR Series and AR Series

Upper Heated Plate

Getting Started Guide

Revision G Issued November 2013

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
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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 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 Upper Heated Plate

About the Upper Heated Plate (UHP)

At the Peltier Plate's temperature range extremes, a vertical temperature gradient may be introduced across the sample, the significance of which will depend on the sample's thermomechanical properties. Although this gradient can be reduced by the use of an upper geometry containing a thermal break, it can only be effectively eliminated if the Peltier Plate and upper geometry are constrained to the same temperature. The Upper Heated Plate (or UHP, shown below) has been developed to allow this and is used in conjunction with the standard Smart Swap™ Peltier Plate.

Figure 1 The Upper Heated Plate (UHP); DHR Series shown.

On the Discovery Series and AR-G2, the system can be configured with the Upper Temperature Sensor (UTS) so that the temperature of the upper plate is measured directly. The UTS consists of a special draw-rod containing a Pt100 temperature sensor. The information from this sensor is transmitted to a remote reading device.

Upper Heated Plate Components

The UHP consists of two main components:

- A fixture that attaches to the rheometer head. This fixture contains electrical heating elements and a coolant channel.
- An upper geometry holder that attaches to the rheometer rotating shaft. The geometry holder contains a heat spreader.

There is no physical contact between the two components (see the figure below). Heating of the UHP is provided by the electrical elements, while cooling is provided by vortex air, water, or other fluid carried in the coolant channel.

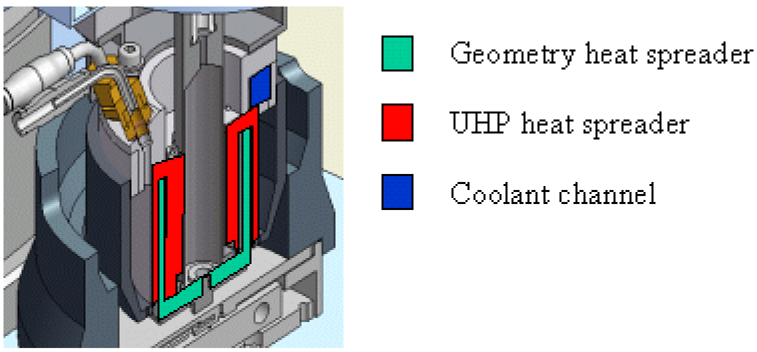


Figure 2 Exploded view of the UHP and upper geometry holder.

Control of the water flow is through a 3-way solenoid valve contained in a Cooling Control Unit (CCU) placed upstream of the Upper Heated Plate. The CCU is also connected to an air supply, allowing purge air to displace water from the coolant channel during heating or at elevated temperatures. If vortex air or fluids other than water are used as coolants, purge air is not required, and the CCU is replaced by a 2-way solenoid.

A Pt100 probe placed within the Upper Heated Plate heat spreader reads the temperature of the Upper Heated Plate. The offset between the read temperature and that of the upper geometry plate is obtained by prior calibration.

An inert gas atmosphere can be produced using the inert gas inlet located between the inlet and outlet coolant ports on the Upper Heated Plate. The inert gas jets are located on the underside of the heating element cover. A protective sample cover and an instrument air bearing clamp are also provided.

System Specifications

Refer to the table below for UHP system specifications:

Table 1: Upper Heated Plate System Specifications

Temperature range plumbed water supply (11°C) low viscosity silicone circulating fluid at -40°C vortex air cooler	 20°C to 150°C -30°C to 55°C -5°C to 150°C
Ramp rate*	15°C maximum
Maximum temperature difference between plates	0.1°C

* 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 Upper Heated Plate

This chapter contains instructions on installing the UHP system on your rheometer. Installation of the UHP includes the following steps:

1 Attaching the UHP to the rheometer

2 Installing the (optional) vortex air cooler

3 Configuring the system for use with cooling water

4 Connecting the geometry holder

5 Firing and connecting the optional UHP / Peltier Manifold

6 Configuring the UHP in TRIOS software

7 Calibrating the UHP

Attaching the Upper Heated Plate to the Rheometer

Follow the steps below to attach the Upper Heated Plate to the rheometer head.

1 Ensure that air (at the correct pressure) is supplied to the air-bearing, and remove the bearing cap. Turn on the rheometer and raise the head to the maximum (use the **Head UP** button located on the instrument control panel).

2 Attach the Upper Heated Plate 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, with the ports for the coolant and inert gas to the left (as shown in the figure below).

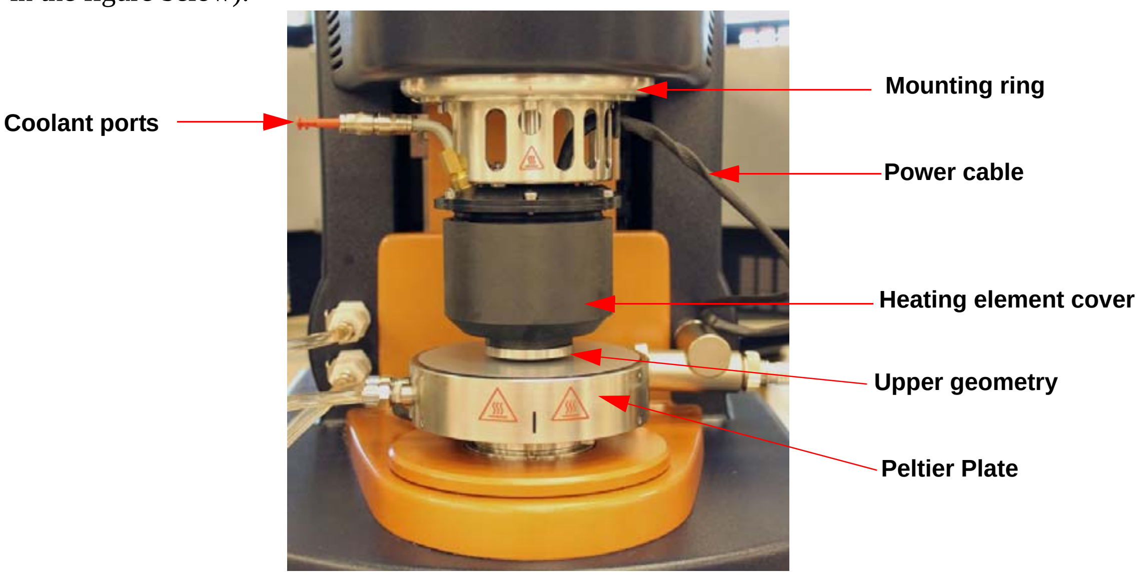
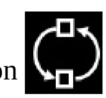


Figure 3 The Upper Heated Plate shown mounted on an AR Series Rheometer.

3 Disconnect the Peltier Plate cable from the Smart Swap™ socket using the **Release** button  on the instrument control panel.

4 Connect the Peltier Plate and Upper Heated Plate cables to the left and right sockets on the Smart Swap Upper Heated Plate adapter respectively (see the figure below). For proper system identification, it is important that this step be completed before step 5.

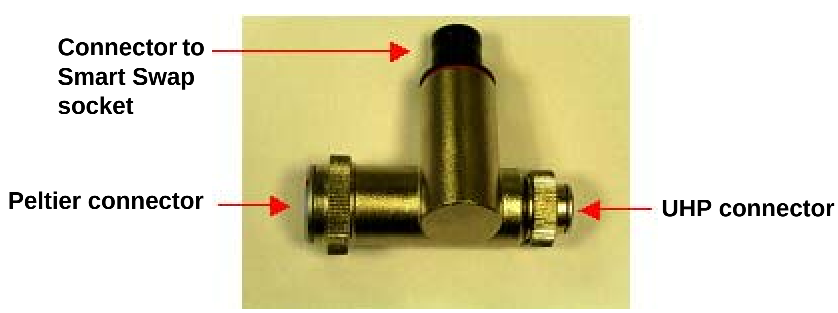


Figure 4 The Smart Swap UHP adapter.

5 Connect the Upper Heated Plate adapter to the Smart Swap socket (see the figure below).

Figure 5 Connection of the UHP adapter to the Smart Swap socket (DHR Series shown).

6 To return the temperature control to Peltier Plate only, remove the adapter from the Smart Swap socket using the **Release** button on the instrument control panel. Remove the Peltier connector from the adapter and plug the connector directly into the Smart Swap socket.

WARNING: Do not remove the heating element cover.

AVERTISSEMENT: Ne retirez pas le couvercle de l'élément chauffant.

Installing the (Optional) Vortex Air Cooler

Follow the steps below to attach and connect the vortex air cooler to the rheometer.

1 Mount the vortex air cooler bracket to the rear of the rheometer using the two screws provided. Screw locations are circled in the figure below. For a DHR instrument, mount the adapter bracket to the rear of the rheometer, and then mount the vortex air cooler bracket to the adapter bracket.

Figure 6 Mounting the bracket on an AR Series Rheometer.

2 Clip the vortex air cooler into the spring clips with the brass muffler extending upward, as shown in Figure 8.

3 Remove the metal push-fit connector from the inlet port on the UHP and fit the Swagelok adapter supplied in the kit. See Figure 7. (Note that once this has been fitted, it cannot be removed. Returning to the push-fit connector will require the supplied adapter.)

4 Connect the black insulated tube between the lower (vertical) outlet of the vortex air cooler and the Swagelok fitting on the UHP inlet, and insulate the exposed metal connections.

5 Cut 800 mm of the white 6-mm O.D. tubing. Connect this tubing between the Upper Heated Plate outlet and the lower bulkhead fitting on the vortex air cooler bracket.

6 Either use the short 6-mm fitting supplied or cut 150 mm of the white 6-mm O.D. tubing and connect to the upper bulkhead fitting on the vortex air cooler bracket. The other end is left open to vent to the atmosphere.

7 Connect white 6-mm O.D. tubing between the outlet of the two-way valve from the CCU unit and the middle (horizontal) inlet of the vortex air cooler.

8 Connect the opposite end of the white 6-mm O.D. tubing used in step 7 to a source of dry compressed air (80 to 100 psi, –30°C dew point or better). An 8-mm “Y”-piece and 8-mm to 6-mm reducer are supplied to break into the rheometer air line before the filter regulator.

9 Connect the event socket on the valve bracket to the **EVENT B** socket on the rear of the rheometer using the cable provided.

Figure 7 UHP Swagelok adapter (AR Series shown).

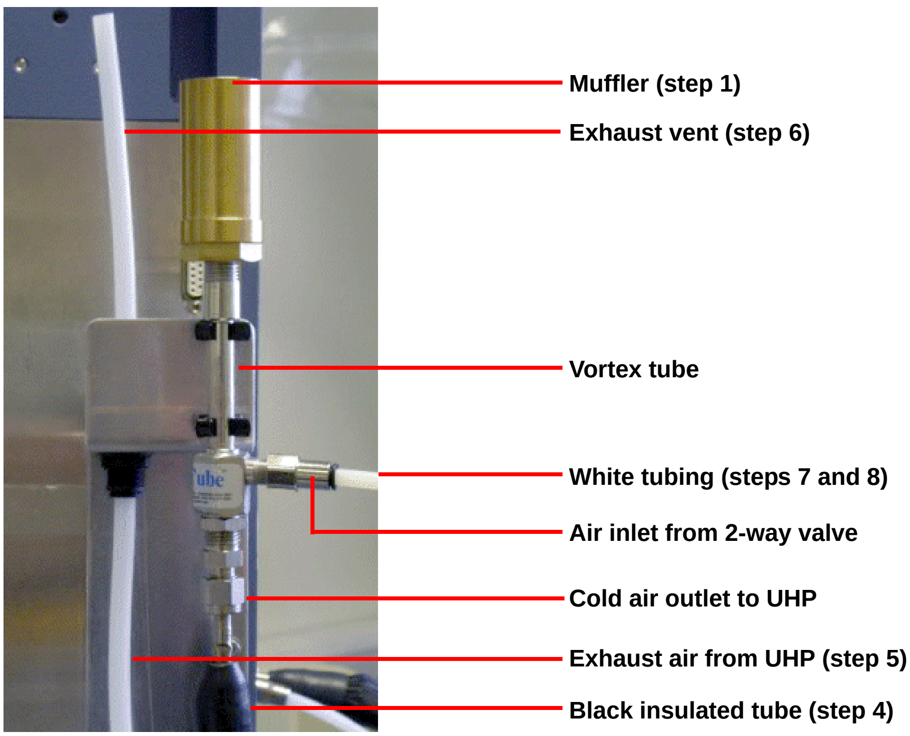


Figure 8 Attaching the vortex air cooler (AR Series shown).

Table 2: Vortex Air Cooler Specifications

Minimum Temperature	Maximum Temperature
-5°C	150°C

NOTE: If you find a reduction in the expected cooling performance, check that there is exhaust air flowing from the white 6-mm O.D. tubing. If there is limited or no air flow, this is an indication that the cold end of the vortex tube is blocked with ice, formed by condensing moisture in the air supply. The tube can be taken apart and ice removed, but the only long-term solution is to supply drier air.

Configurations for the Cooling Water

The minimum temperature and the cooling rate attainable on the Upper Heated Plate will depend on the temperature, flow rate and heat capacity of the circulating fluid. In general, provided that the flow rate is adequate, the minimum temperature will be about 5°C above that of the circulating fluid at the inlet, although this will depend on the ambient conditions. The standard configuration is with water as the circulating fluid, in which case mains water or a general laboratory circulator can be used.

It is recommended that separate sources should be used for the cooling water supplied to the Peltier Plate and the Upper Heated Plate, as the pulsing of the cooling water can influence the instrument normal force reading. However, the same supply may be used for both units, provided that sufficient pressure is available to ensure adequate flow through both (for example from an FP50-MS fluid circulator available from Julabo GmbH; mains water supply is also normally suitable). Some possible configurations are shown below.

Important: For efficient operation, the Peltier Plate and Upper Heated Plate should be connected in parallel, NOT in series, if the same water supply is used for both.

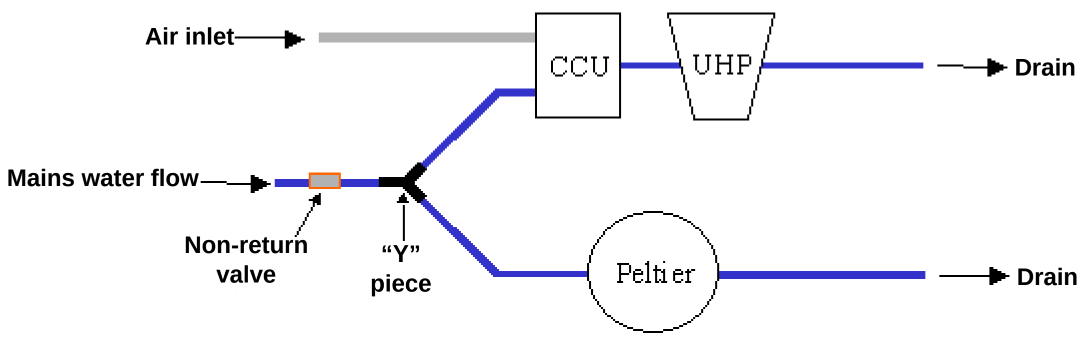


Figure 9 Cooling water configuration 1: Mains water supplying both Peltier and Upper Heated Plate.

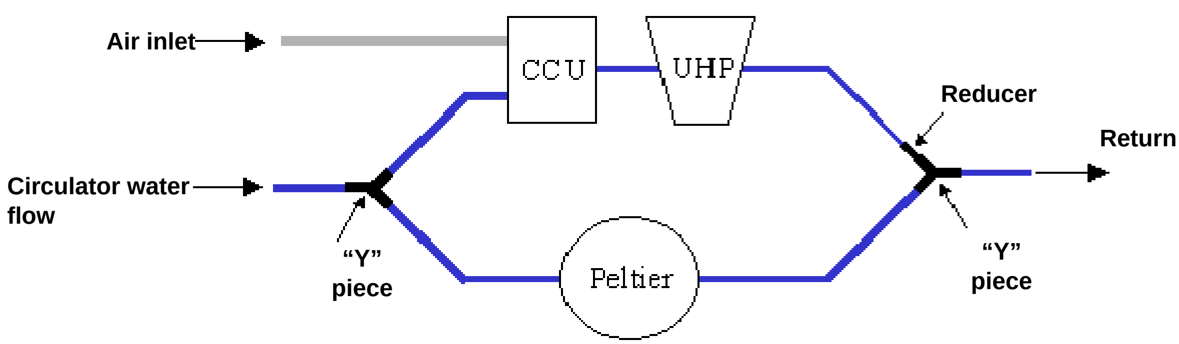


Figure 10 Cooling water configuration 2: Fluid circulator supplying both Peltier and Upper Heated Plate.

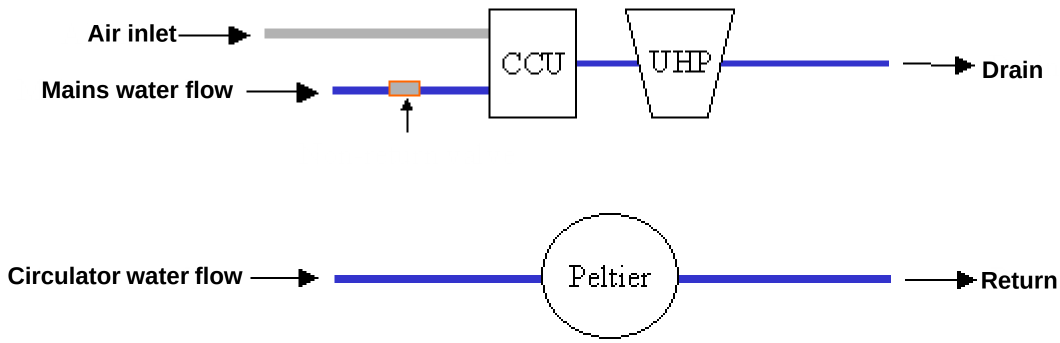


Figure 11 Cooling water configuration 3: Fluid circulator supplying Peltier, mains water supplying Upper Heated Plate.

Alternative configurations, not shown here, are for the Upper Heated Plate and Peltier to be supplied by separate fluid circulators, and for the Upper Heated Plate to be supplied by a fluid circulator, the Peltier by mains water. The non-return valve is not required for either of these configurations.

Connecting the Cooling Control Unit

This unit (shown in the figure below) may be free standing, or wall mounted using the clearance holes on top of the unit.

Figure 12 The Cooling Control Unit.

1 Connect the air supply to the **GAS IN** port on the CCU using the 8 mm outer diameter tubing (white). If it is necessary to split the air line to provide a source for both the instrument air bearing and the CCU, this should be done upstream of the instrument filter regulator system.

2 Connect the water supply to the **LIQUID IN** port on the CCU using the 6 mm outer diameter tubing (blue). If mains water is used as the supply, then the non-return valve (shown in the figure below) should be placed in the line upstream of the CCU.

Important: Note the direction of flow through this valve.

Figure 13 Non-return valve (for use with mains water supply only; note the direction of flow through the valve).

3 Connect the **GAS / LIQUID** outlet port on the CCU to the **Coolant Inlet** port on the Upper Heated Plate using the 4 mm outer diameter tubing (blue) and the 4 mm to 6 mm adapter provided. Refer to Figure 14.

4 Connect the **Coolant Outlet** port on the Upper Heated Plate to **drain** if mains water is the supply, or to **return** if a fluid circulator is used. Use the 4 mm outer diameter tubing (blue) for this port. A 4 mm to 6 mm adapter and 6 mm "Y" piece are provided for the connection to the fluid circulator. See the figure below.

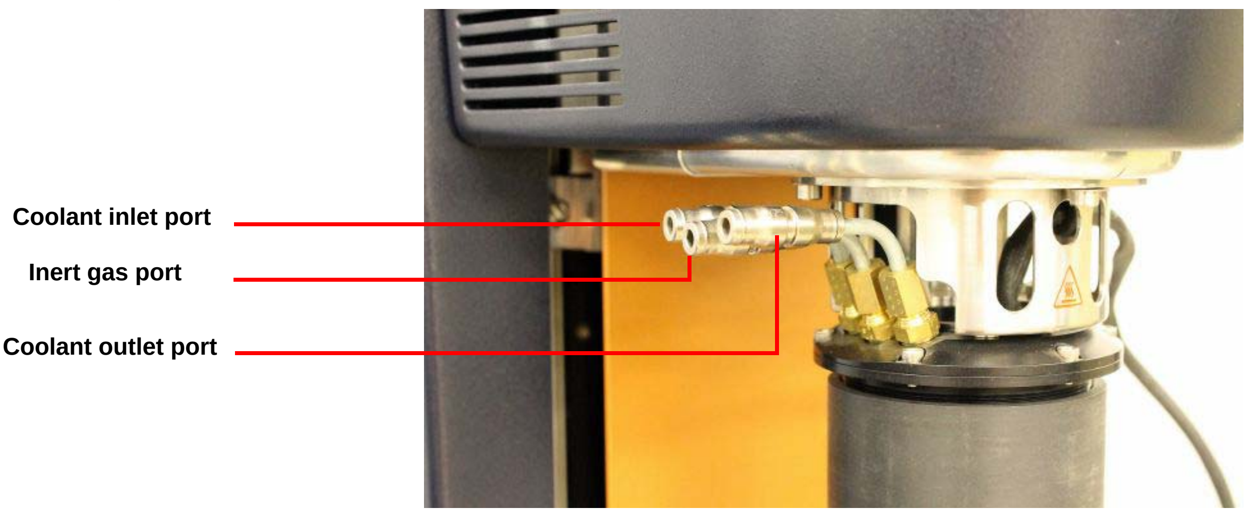


Figure 14 Coolant and inert gas connections for the UHP (DHR Series shown).

5 Connect the **EVENT** socket on the CCU to the **EVENT B** socket on the rear of the rheometer using the cable provided.

6 Set the purge air flow rate to 1 L/min. Note that the reading is taken from the center of the float. To set the flow rate, it may be necessary to raise the temperature of the Upper Heated Plate using TRIOS Software to ensure continuous air flow.

Using Circulating Fluids Other Than Water

For low temperatures, Use circulating fluids other than water for low temperatures. These should be fluids of the silicone type, as recommended by the supplier of the fluid circulator. A separate kit is available for use with these fluids.

WARNING: Flammable fluids such as ethanol or mineral oils should NOT be used with the Upper Heated Plate. Circulating fluids should NOT be used outside the ranges given by the supplier.

AVERTISSEMENT: Les liquides inflammables comme l'éthanol ou les huiles minérales ne doivent PAS être utilisés avec la plaque supérieure chauffée. Les fluides de circulation ne doivent PAS être utilisés en dehors des plages indiquées par le fournisseur.

Silicone fluids are usually higher in viscosity than water, and the required flow rates cannot be achieved with the standard CCU described above. The special low temperature kit should replace this. As when water is used as the circulating fluid, it is suggested that separate sources should be used for the cooling fluid supplied to the Peltier Plate and the Upper Heated Plate. Then water may be used for the Peltier, and a silicone fluid for the Upper Heated Plate, for example. However, the same supply may be used for both units, provided that sufficient pressure is available to ensure adequate flow through both. Note that if a single supply is used, the Peltier and Upper Heated Plate should always be connected in parallel, never in series.

1 Connect the flow port on the fluid circulator to the inlet of the 2-way CCU valve using the 6 mm outer diameter (blue) tubing provided.

2 Connect the outlet from the valve to the Upper Heated Plate inlet, and the outlet from the Upper Heated Plate to the circulator return port using the 6-mm outer diameter tubing (blue). Note that when silicone fluids are used as coolants, the air purge on the Upper Heated Plate is not required.

3 Connect the **EVENT** socket on the CCU to the **EVENT B** socket on the rear of the rheometer using the cable provided.

The table below shows minimum and maximum temperatures for the Upper Heated Plate using circulating fluids available from Julabo GmbH, with an FP50-MS fluid circulator supplied by the same company.

Table 3: UHP Temperature Ranges, by Circulating Fluid

Circulating Fluid	Minimum Temperature (°C)	Maximum Temperature (°C)
Water	5	150
Thermal HY	-30	55
Thermal H5S	-20	105
Thermal H10S	-10	150

Connecting and Disconnecting the Geometry Holder

WARNING: The Upper Heated Plate fixture, upper geometry holder, and the upper geometry may be hot. Ensure that these components are cool before attempting to remove or replace the upper geometry holder.

AVERTISSEMENT: La plaque supérieure chauffée, le support de géométrie supérieur et la géométrie supérieure peuvent être chauds. Assurez-vous que ces composants sont froids avant d'essayer de retirer ou de remplacer le support de géométrie supérieur.

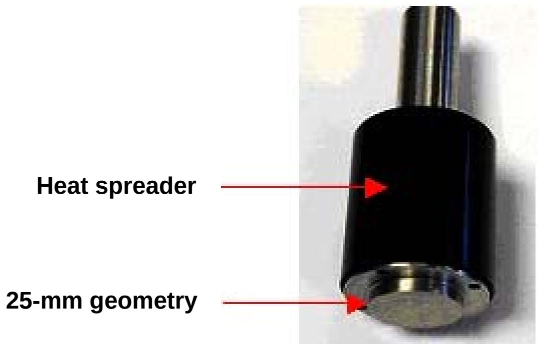


Figure 15 Upper geometry holder (shows the cylindrical heat spreader with a 25 mm diameter geometry in place).

Connecting the Geometry and Holder

To connect the upper geometry and holder follow these steps:

1 Raise the instrument head fully using either TRIOS software or the **Head UP** button located on the instrument control panel.

2 Attach the geometry to the holder using the attaching tool provided. (This tool cannot be used with the 40-mm diameter geometry, which can attached to the holder by hand.) If the Upper Temperature Sensor (UTS) is being used with the Discovery Series or AR-G2, the geometry and upper geometry are a single assembly.

3 When the geometry is in place, carefully insert and position the holder within the Upper Heated Plate, and connect to the instrument shaft by rotating the drawrod. For Upper Heated Plate geometries, a backoff distance of 120,000 µm is recommended.

4 Use 1.448 x 10^{-3} rad/Nm for the geometry compliance, unless other information is available.

Removing the Geometry and Holder

To remove the upper geometry and holder from the rheometer follow these steps:

1 Raise the instrument head fully using either TRIOS software or the **Head UP** button located on the instrument control panel. Grasp the holder firmly, and unscrew from the instrument shaft by rotating the drawrod.

2 Lower the geometry holder carefully until it is clear of the Upper Heated Plate.

3 When the geometry holder is free of the instrument, the geometry can be removed from the holder using the geometry attaching tool provided, if necessary. If the Upper Temperature Sensor (UTS) is being used with the Discovery Series or AR-G2, the geometry and upper geometry are a single assembly.

Fitting & Connecting the Optional UHP/Peltier Manifold

The UHP/Peltier manifold provides an effective, tidy, solution for the network of interconnecting tubing and cables when the UHP is to be used in the temperature range 5 to 90°C. Using water at 2°C as the circulation fluid makes it an ideal choice for combining with the stepped Peltier Plate and UHP in the dry asphalt configuration.

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

2 Looking at the rear of the rheometer:

a Connect the fluid supply from your circulator to the push fittings on the left-hand side of the manifold using the 6mm tubing provided.

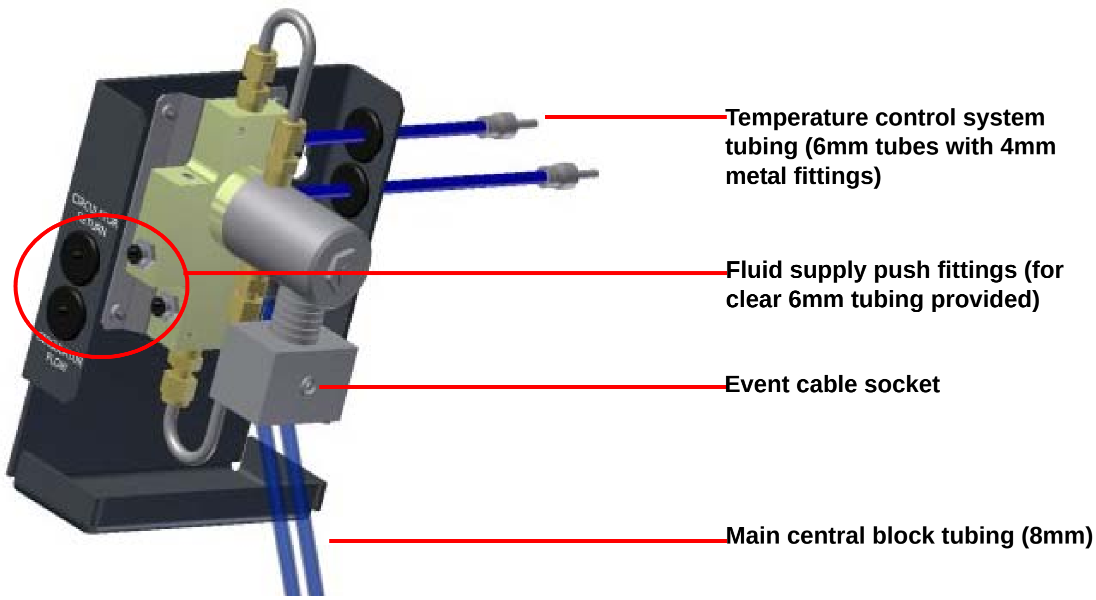


Figure 16 Manifold fittings and tubings.

b Connect the temperature control system tubes exiting the manifold on the top right-hand side to the UHP.

c Connect the tubes exiting from the main central block to the Peltier flow and return push fittings on the rheometer.

NOTE: It is not necessary to change the grommets and fittings on the manifold in order to make connections; use the supplied adaptors to connect the tubing as needed.

NOTE: It may be necessary to insulate long tubing runs with pipe wrap to achieve the minimum temperature specification.

d Connect the Event cable to the Event cable socket on the manifold and then connect the event cable to **Event B** on the back of the instrument.

Figure 17 Manifold mounted to AR Series Rheometer.

Table 4: UHP Specifications for use with Optional Manifold

Fluid Source	Minimum Temperature	Maximum Temperature
ThermoCube @ 2°C	5 °C	90°C

Configuring the UHP

The temperature of the Upper Heated Plate is controlled through the instrument firmware. For the best performance, the control algorithm requires accurate information concerning the thermal properties of the Upper Heated Plate and the cooling fluid. In TRIOS Software, click the TRIOS icon **> Options > AR > Temperature**.

Refer to *Configuring the Upper Heated Plate* TRIOS Help topic for additional information.

Calibrating the Upper Heated Plate

The temperature of the UHP is read from a probe positioned within the UHP heat spreader. The probe should be as close to the upper geometry as possible, although not in physical contact with it. The temperature of the Peltier Plate is read from a probe positioned in thermal contact with the plate, as close to the surface as possible. The temperature reported by the software is that of the Peltier probe. For best performance, the UHP probe should be calibrated to the temperature of the upper geometry plate.

NOTE: Calibration should always be performed on installation of the Upper Heated Plate, and at least annually thereafter. The calibration routine may take several hours, and it is more efficient to perform a single calibration with more points, rather than several calibrations with fewer points.

During the automatic calibration routine, a heat flow sensor determines the temperature gradient between the Peltier Plate and the upper geometry. The gradient is reduced to within preset tolerances by adjusting the temperature of the UHP, while the temperature of the Peltier Plate is held constant.

After each adjustment, a user-defined stability criterion is applied and, once temperature stability is achieved, comparison is made with the gradient tolerance. When the gradient tolerance condition is satisfied, the temperature value is accepted.

The procedure is repeated for a number of points over a range set by the user. When the calibration routine is complete, the temperature values for the upper geometry determined by the calibration are compared with those reported by the UHP probe to obtain the appropriate offset and span values.

For instructions on calibrating the UHP, refer to *AR Rheometer Temperature Calibrations* TRIOS Help topic.

Chapter 3:

Use and Maintenance

This chapter contains information regarding the use of the UHP, and how to attach the bearing clamp when the UHP is not in use.

Using an Inert Gas Atmosphere

Many samples experience oxidation at elevated temperatures—an atmosphere of inert gas such as nitrogen or argon can be used to prevent this. The gas supply should be regulated to less than 40 psi (2.8 bar) before connection to the Upper Heated Plate. A gas flow meter (not supplied) should be used to set the gas flow rate.

1 Connect the gas supply to the inlet port on the gas flow meter.

2 Connect the outlet port on the flow meter to the inert gas inlet port on the Upper Heated Plate using 4-mm outer diameter tubing (white) and the connector provided.

3 Set the inert gas flow rate at 1 L/min. If the gas flow rate is set too high, temperature control of the Upper Heated Plate may be affected.

Using the Sample Cover

Some samples are affected by drafts and general air flow, which can cause drying at the sample edge. To avoid this, a protective sample cover is provided. The cover should be placed in the up position during sample loading and trimming: the cover is held in this position by a bayonet fitting that attaches over the coolant connectors. The cover should be used in the down position during the experimental run.

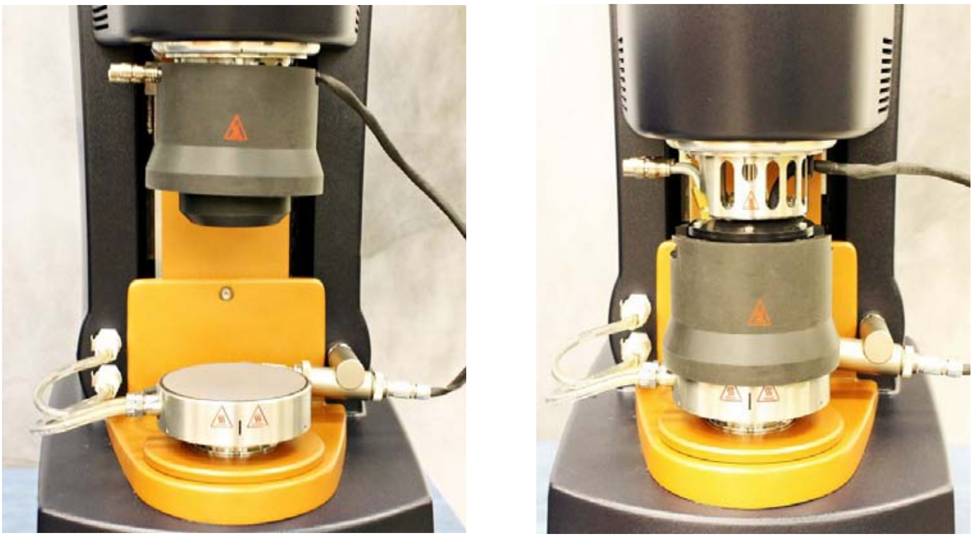


Figure 18 Sample cover in up position (left) and down position (right) on DHR Series Rheometer.

WARNING: The sample cover may be hot. Ensure that it is cool before attempting to raise or remove it.

AVERTISSEMENT: Le couvercle de l'échantillon peut être chaud. Assurez-vous qu'il est froid avant d'essayer de le soulever ou de l'enlever.

Using the Upper Heated Plate for Asphalt Testing

The UHP combined with the stepped Peltier Plate is the temperature system used for the "Dry Asphalt System." Information on running asphalt binder tests and performing additional system calibrations are covered in the Asphalt Submersion Cell Manual.

NOTE: The standard asphalt configuration for all instruments uses the holder with removable geometry.

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