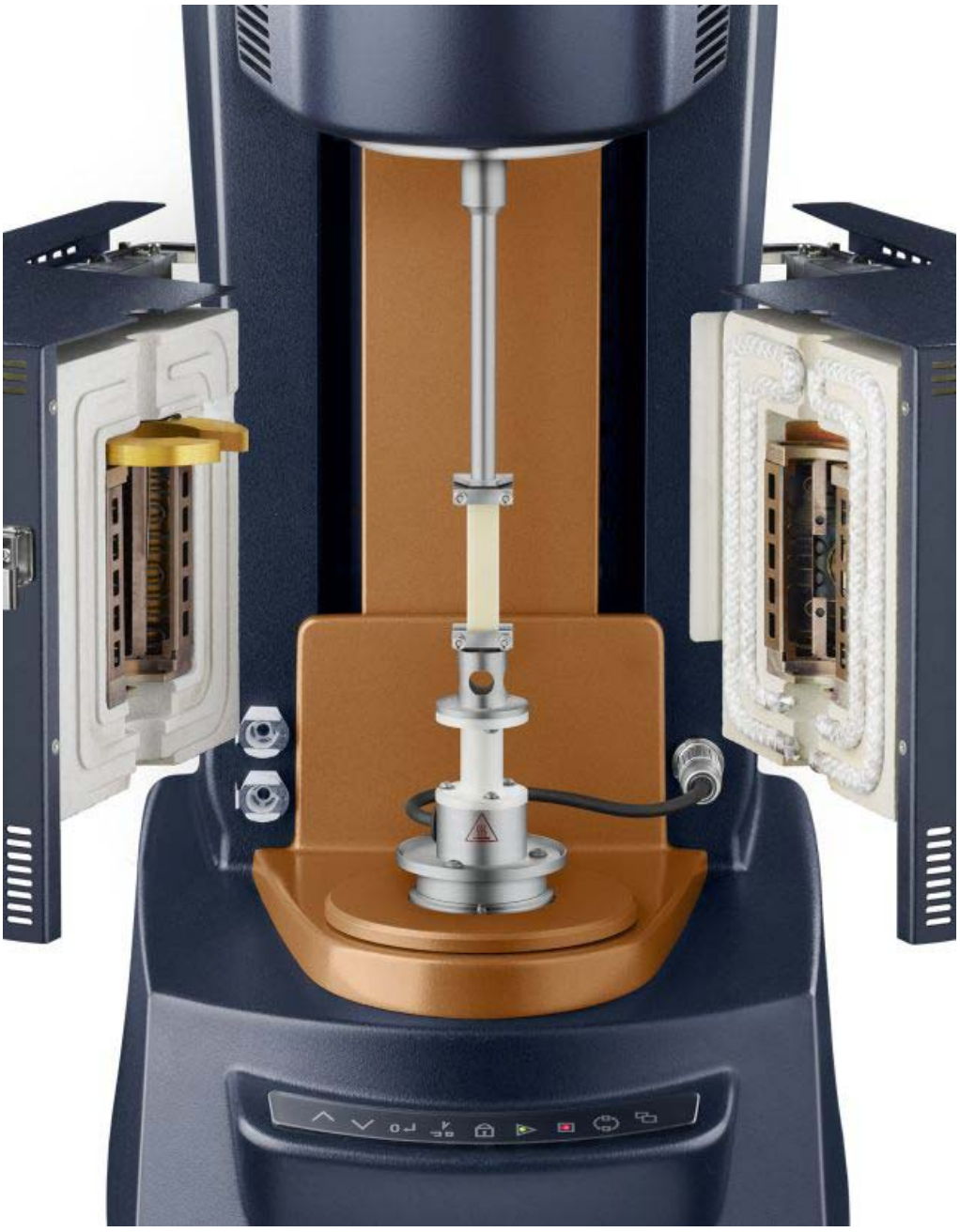
DHR Series and AR Series

Environmental Test Chamber



Getting Started Guide

Revision E **Issued May 2014**

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
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- Other Trademarks
- TA Instruments End-User License Agreement
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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.

Table of Contents

Chapter 1:

Introducing the Environmental Test Chamber

About the Environmental Test Chamber (ETC)

The ETC is an add-on unit for the DHR/AR-G2/AR 2000ex that enables it to characterize solid samples in torsion. To work on composites/polymers in torsion requires a temperature-controlled chamber for environmental control of the sample, plus specially designed sample clamps. The ETC can also be used for conventional rheology measurements with the appropriate geometries.

Figure 1 ETC mounted on DHR.

ETC Components

The ETC chamber is mounted onto a metal plate that is secured to the back of the rheometer. The DHR ETC has dowel pins for alignment that also provide support while the unit is secured in place. Handles at the bottom of each door are provided for lifting and positioning.

The oven is made up of two halves (left and right as viewed from the front), with each half containing a specially designed heating element with a thermocouple welded to it (see Figure 2). Various interlock devices are fitted for safety purposes. These devices detect the position of the doors. The Status Bar in TRIOS software reports the current state: **open**, **partly open**, or **closed**. Temperature control is only active when the doors are closed. Note: You will not be able to move the head up unless the doors are fully open.

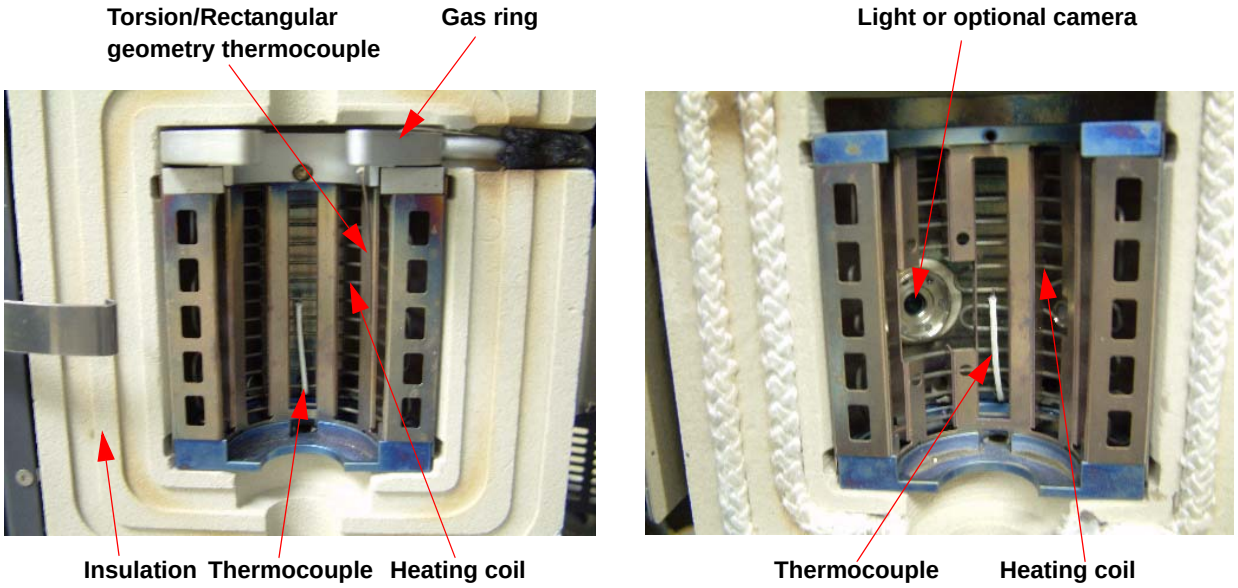

Figure 2 ETC interior, left and right sides.

The left-hand door also incorporates the gas ring, which allows stirring air and liquid nitrogen to be fed into the chamber. The control electronics are contained within the main electronics of the ETC.

The ETC Viewer

WARNING: After ETC use, do not turn off the cooling air to the ETC Viewer until the unit has returned to room temperature. Doing so could damage the lens assembly and/or camera chip.

AVERTISSEMENT: Après l'utilisation de l'ETC, n'arrêtez pas le passage de l'air de refroidissement vers l'afficheur ETC tant que l'appareil n'est pas revenu à la température ambiante. Cela pourrait endommager l'ensemble des lentilles et/ou la puce optique intelligente.

The ETC Viewer is as an option that can be used with the rheometer for the following purposes:

- Viewing the edge of plates and cones and the torsion sample
- Providing streaming video
- Capturing images with data point (not fast sampling)
- Viewing point image in TRIOS software and pseudo playback

The software provided with the ETC Viewer works with any USB camera so a web cam can be easily used for Peltier tests. The viewer assembly is air cooled to allow it to perform over whole temperature range of the ETC (–160 to 600°C).

System Specifications

Refer to the table below for ETC system specifications:

Table 1: ETC Specifications

Temperature range no cooling LN2 cooling ACS-3 cooling	 50°C to 600°C –160°C to 600°C -85°C to 600°C
Typical ramp rate	Maximum ramp rate up to 60°C/min[1]
Internal resolution	0.02°C

1. Ramp Rate: The maximum sustainable ramp rate will depend on a number of factors particularly the start and end temperature. To determine the maximum sustainable heating/cooling rate, perform the following test and analysis:

   a. 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.

   b. 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 ETC

This chapter provides information on how to install and set up the Environmental Testing Chamber (ETC).

Installing the ETC Assembly on the DHR Series

Follow the instructions below to install the ETC on a DHR Series Rheometer.

Mounting the Test Chamber

1. Turn on the rheometer and move the rheometer head up to the maximum height. Use the **Head Up** button located on the instrument control panel.
2. Turn off the power to the rheometer control box.
3. The ETC is most easily fitted with the rear of the instrument facing you. It may be necessary to disconnect the power and signal cables to rotate the instrument to provide access.
4. Locate the four dowel holes on the rear of the instrument, as well as the four dowel pins on the ETC bracket. See the figure below.

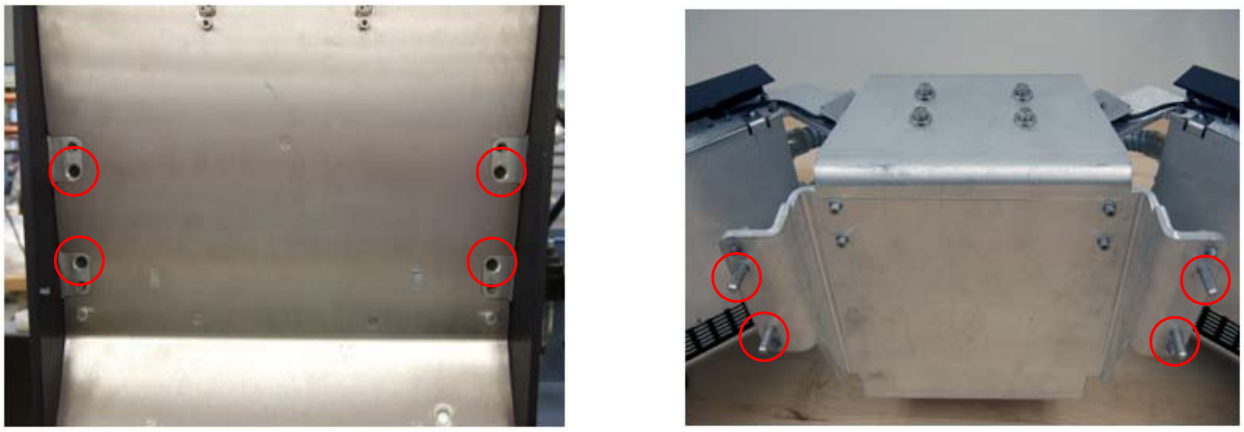

Figure 3 Dowel holes on rear of instrument (left image); dowel pins on ETC bracket (right image).

5 Lift the ETC with the doors fully open using the handles, as shown below.

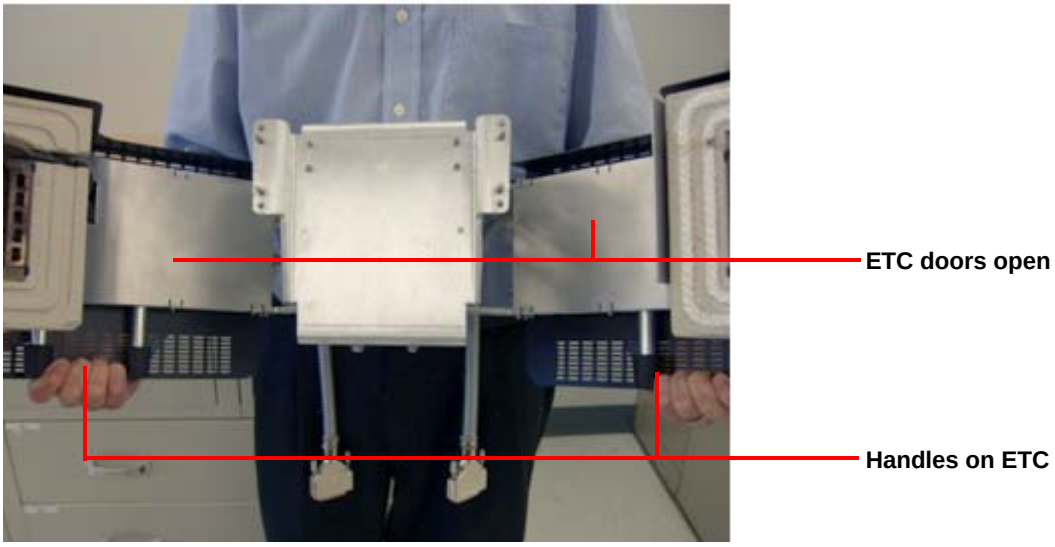

Figure 4 Lifting the ETC door.

6 Insert the dowel pins on the ETC into the dowel holes on the rear of the instrument, as shown in Figure 5.

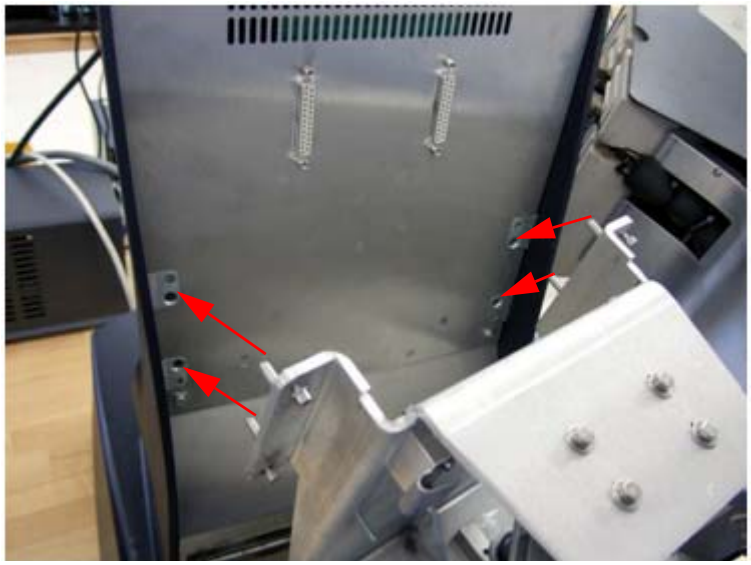

Figure 5 Mounting the ETC on the rear of the instrument.

7 Once in position with the doors still fully open, the ETC will self-support on the dowel pins.

Figure 6 ETC mounted on back of instrument with doors fully open.

8 Screw the four +captive screws into the casting to secure the ETC to the instrument.

Figure 7 Securing the ETC to the instrument.

9 Plug in the two ETC D-type connectors into the **Attachment Left** and **Attachment Right**.

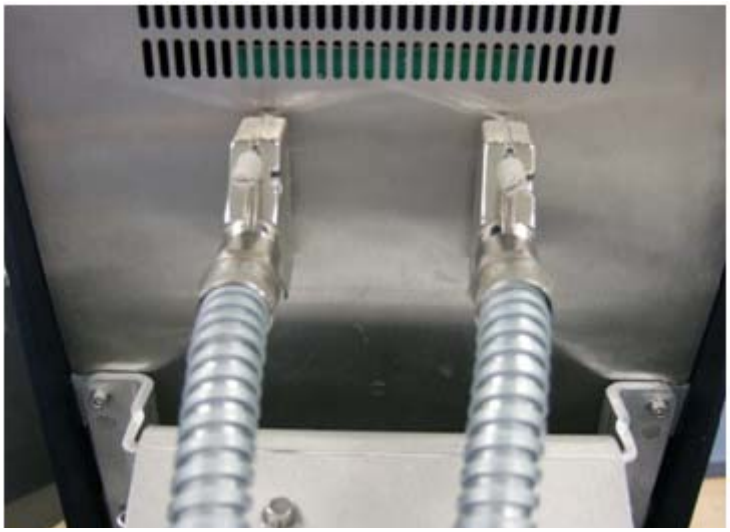

Figure 8 Attachments installed on rear of instrument.

10 Open the ETC oven door to gain access to the Smart Swap™ mounting.

Removal of the ETC is the reverse of the preceding steps. Note that you can leave the ETC in place and still use other Smart Swap temperature systems.

Installing the ETC Assembly on the AR Series

Follow the instructions below to install the ETC on an AR-G2 or AR 2000ex.

Mounting the Test Chamber

1 Turn on the rheometer and move the rheometer head up to the maximum height. Use the **Head UP** button, located on the instrument control panel.

2 Fit the air-bearing clamp to the rheometer.

3 Turn off the power to the rheometer control box.

4 Ensure that the two top screws (**A** and **B** in the figure below) are fitted with washers and are located in place—but make sure that they are almost totally unscrewed (two turns in).

Figure 9 Mounting screws.

5 Open the ETC oven and then use the handles on the oven doors to lift it onto the two top screws. Lightly

tighten these screws.

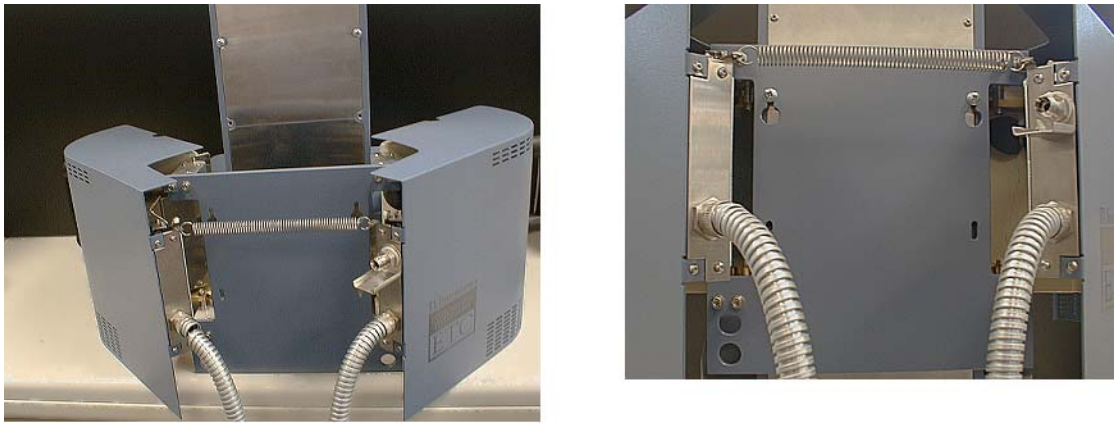

Figure 10 The ETC open.

6 Insert the final two screws (**C** and **D** in Figure 9).

7 Adjust the position of the ETC on the screws and then tighten all four screws.

8 Check the adjustment and adjust if required by loosening the screws and shifting the position of the ETC on the rheometer.

9 Connect the two cables on the ETC to the attachment connectors on the rheometer as shown in the figure below.

10 With the ETC oven doors open, gain access to the Smart Swap™ mounting. Press the **Release** button  on the instrument front panel. A continuous green light indicates that the attachment can be fitted.

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

Removal of the ETC is the reverse of the preceding steps. Note that you can leave the oven in place when you wish to use the one of the Peltier systems.

Installing the Lower Assembly

1 Fit the lower attachment, ensuring it is aligned correctly. See the figure below.

Figure 11 Fitting the lower attachment.

2 Connect the accessory cable from the lower attachment to the rheometer, as seen below.

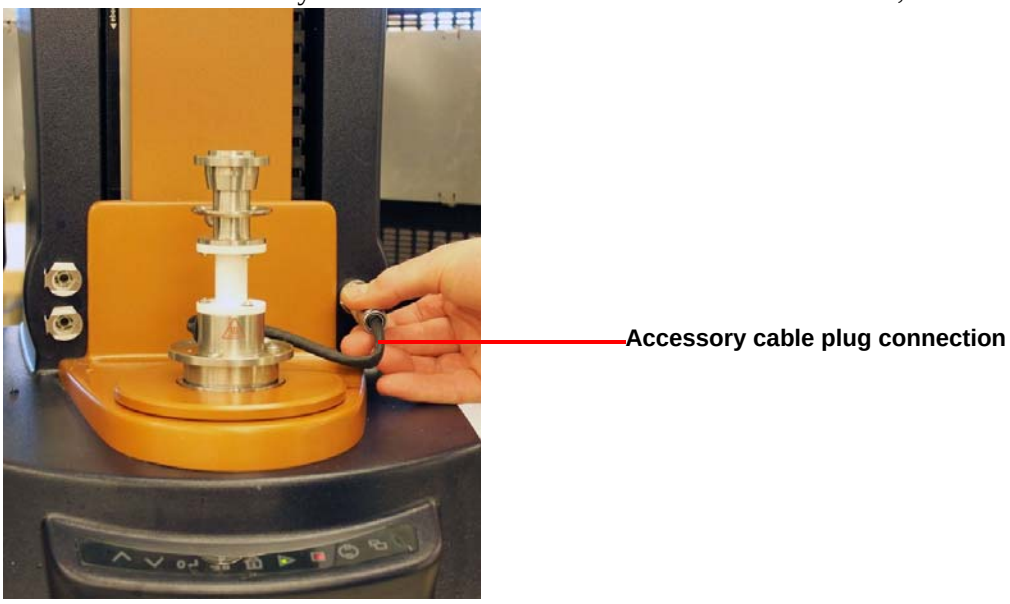

Figure 12 Connecting the accessory cable from the lower attachment to the rheometer.

3 Close the oven and ensure that no part of the doors touch any part of the lower fixture. No change should be observed in the axial force when the doors are closed. Adjust the position of the ETC again, if required.

4 Attach the upper geometry, again making sure that no parts are touching the fixture, and adjust the ETC if necessary.

5 If you plan to use the liquid nitrogen option or ACS-3 air chiller with the ETC, skip the following steps and proceed to "Installing Low Temperature Accessories" on page 21. Otherwise, continue with the instructions below.

6 Connect the purge gas to the rheometer, as shown in Figure 13. If you have a suitable supply of nitrogen gas (2 bar minimum pressure, nominal 10 L/min flow rate) it is recommended that you connect the feed gases to the ETC as shown in Figure 15. Otherwise, connect as shown in Figure 14 below.

CAUTION: The reducing valve is factory-set to 10 L/min and should not be adjusted.

MISE EN GARDE: Le soupape de réduction est réglée en usine à 10 L/min et ne doit pas être ajustée.

Figure 13 Connecting the purge gas.

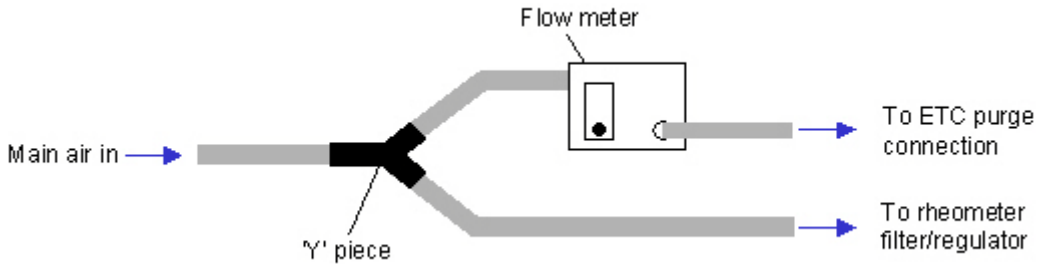

Figure 14 ETC connections using air as the agitation gas.

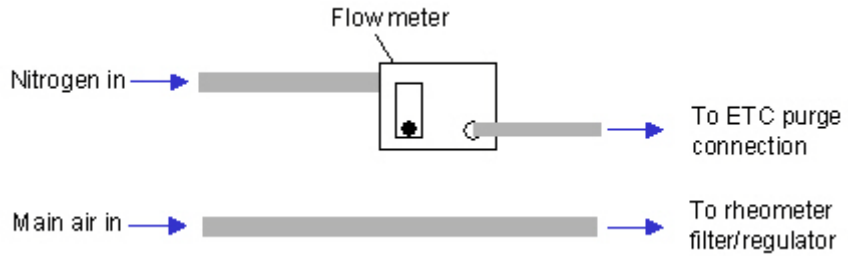

Figure 15 ETC connections using nitrogen as the agitation gas.

Installing Low Temperature Accessories

In order to operate the ETC at temperatures below ambient and also to facilitate rapid cooling, (optional) low temperature accessories can be used. These work by supplying a controlled flow of cold gas to the oven.

Follow the installation procedure in the previous section up to step 4. Then use the following additional steps to complete the installation of the liquid nitrogen option, or refer to the ACS-3 Getting Started Guide.

1 Connect the Event cable from the CCU flow control assembly to the **Event A** connection on the rheometer.

Figure 16 Event connection.

2 Ensure that the cryogenic system has been installed as directed in the instructions supplied by the manufacturer.

3 Connect the tap and solenoid valve unit to the liquid nitrogen outlet on the Dewar, and the unit to the **Liquid in** port on the flow meter assembly using the long flexible hose provided. Connect the **Gas/ Liquid Out** port on the flow meter assembly to the purge gas port on the ETC using the short flexible hose provided, as shown in the figure below.

Figure 17 Connecting the hoses (DHR Series shown).

The electrical and gas/ liquid connections for the ETC low temperature assembly are shown in Figure 12.13.

4 Connect a gas feed to the **Gas In** port on the flow meter assembly using the 8-mm internal diameter white nylon tubing provided. If the main air supply is used as the feed gas, the line should be split *upstream* of the flow meter assembly and rheometer filter/regulator.

5 Connect the cable from the **Liq** connector on the flow assembly to the solenoid valve on the cryogenic system.

6 Set a pressure of 15 to 20 psi on the Dewar system.

7 Open the control valve approximately two full turns*.

8 Set a flow rate of 10 L/min on the flow meter assembly. Make sure the oven is closed.

*The exact setting depends upon the required operating conditions for the ETC as well as the type of cryogenic cooling system used. Additional information on this setting can be found in Chapter 3.

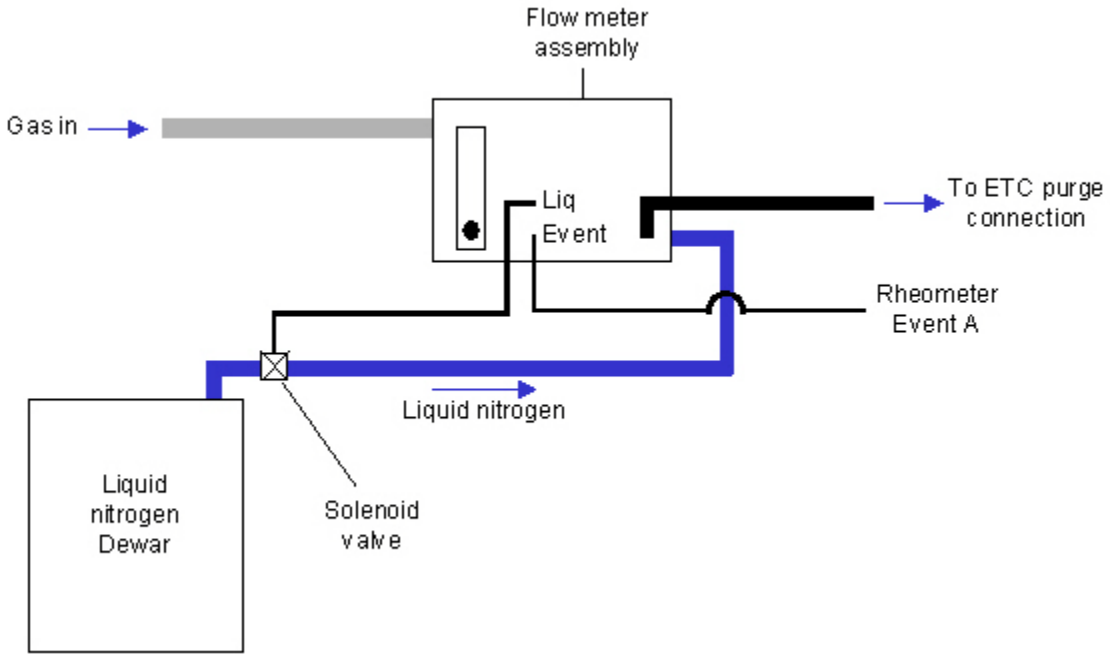

Figure 18 Electrical and gas/liquid connections for the ETC Low Temperature assembly (The electrical connections are shown as the narrow black lines.)

Installing/Removing the ETC Viewer

All ETCs are shipped in a state which is prepared to install the ETC Viewer. Insulated plugs fill the viewer and secondary lighting cavities that are needed for the viewer. The viewer will generally be fitted by a TA Service Engineer, but if the unit needs to be removed for return to a TA service shop for repair, follow these instructions.

WARNING: Make sure ETC is at ambient temperature before attempting this procedure.

AVERTISSEMENT: Assurez-vous que l'ETC est à température ambiante avant d'essayer cette procédure.

NOTE: The pictures shown are for the AR Series, but the process is essentially the same for the DHR Series.

1 Turn off power and air.

2 Remove the two 3-mm hex head screws shown in the image below.

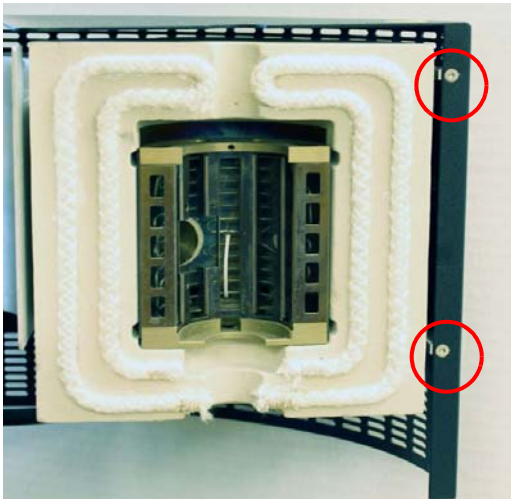

Figure 19 Removing 3-mm hex screws.

3 Loosen the two 3-mm hex head screws shown in Figure 20. This will allow the cover to be removed, as shown in Figure 21.

Figure 20 Removing 3-mm hex screws.

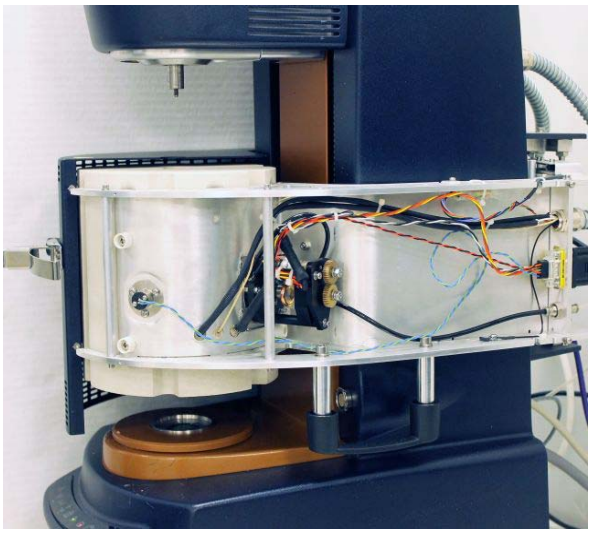

Figure 21 ETC with covers removed.

4 Remove the three 3-mm hex head bulkhead screws holding the secondary lighting in place and remove the unit. Fit the insulated plug in its place.

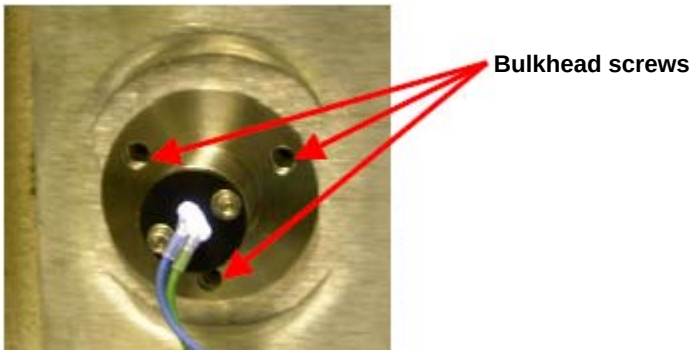

Figure 22 Removing the bulkhead screws.

5 Remove the three 3-mm nuts holding the main viewer module in place and remove the unit. Fit the insulated plug in its place.

NOTE: The location of the stand-offs is shown in the figure below. The actual stand-offs are not visible in the picture.

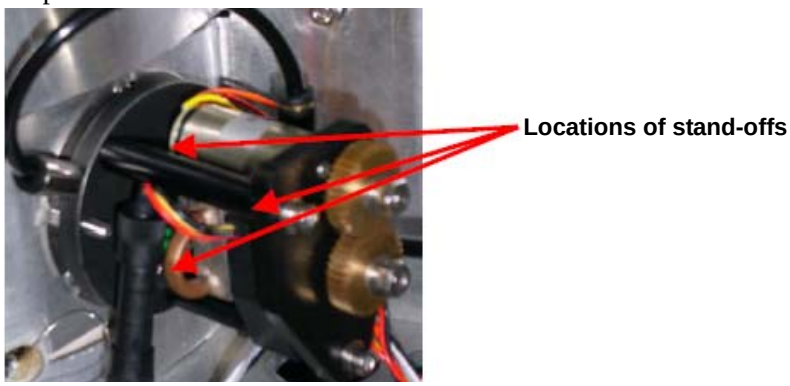

Figure 23 Standoff locations.

6 Disconnect the signal/power connector and cooling air supply from the inside of the back panel shown in the figure below.

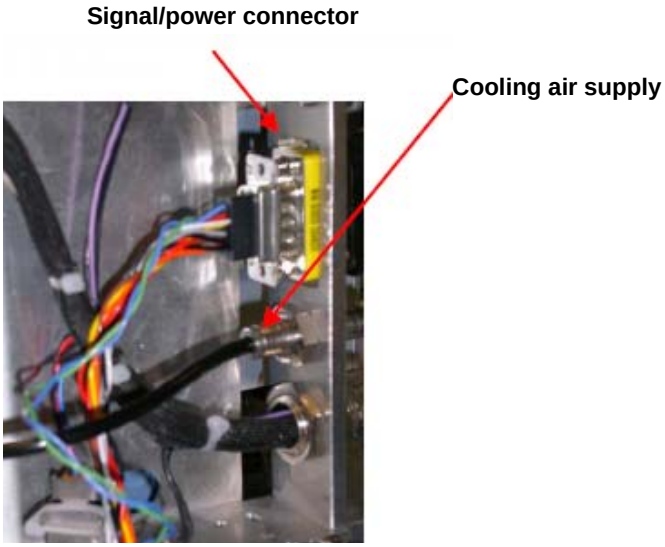

Figure 24 Disconnecting power.

7 The complete unit can now be removed and packed in its original packing material.

8 Follow the instructions in reverse for fitting the viewer module.

Connecting the Camera Viewer

The picture below shows the viewer control box and connections.

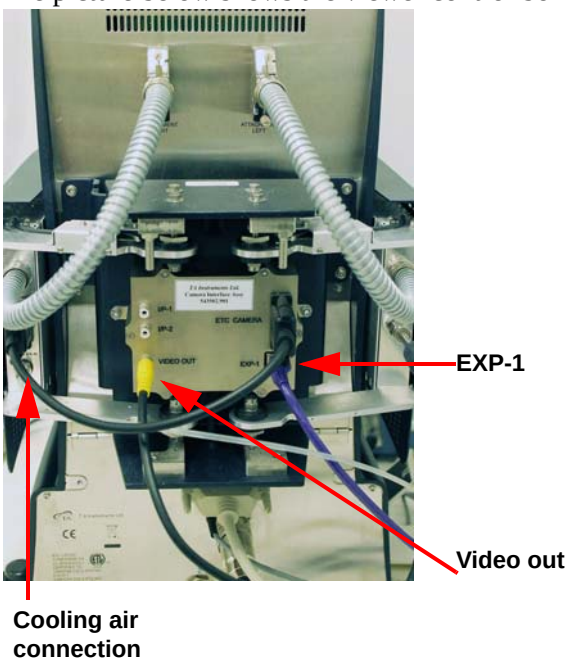

Figure 25 Viewer control box and connections.

1 Place a “T” connector in the main air line to the rheometer, before the filter regulator, and then feed through the supplied flow meter at 15 L/min. This is for the cooling air supply.

2 Connect **EXP-1** on the control box to **EXP-1** on the rear of the main electronics box. See the figure below.

Figure 26 Rear of main electronics box.

3 Connect the video out to a USB adapter, which is in turn connected to a USB port on the control PC. Refer to the installation instructions provided by the original equipment manufacturer (OEM supplier) for installing the driver needed for this device.

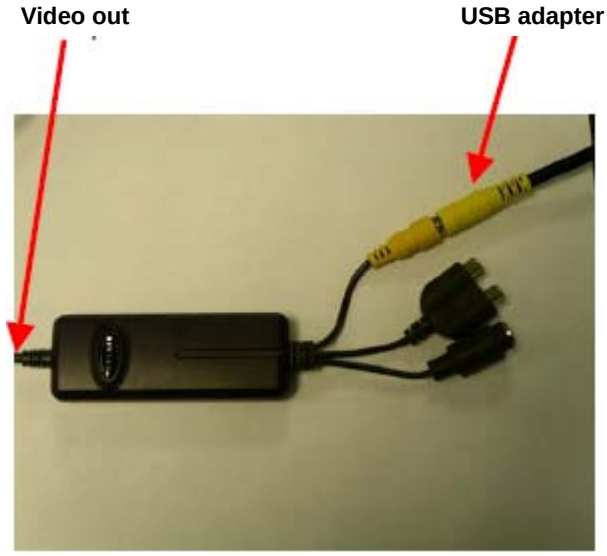

Figure 27 USB adapter assembly.

ETC Viewer Schematics

The following figures provide schematics of the various components of the ETC Viewer.

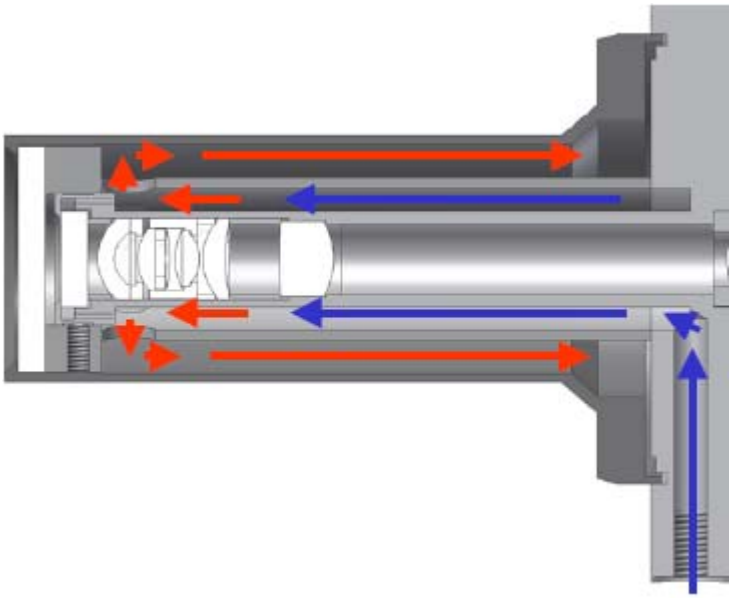

Figure 28 Cooling schematic.

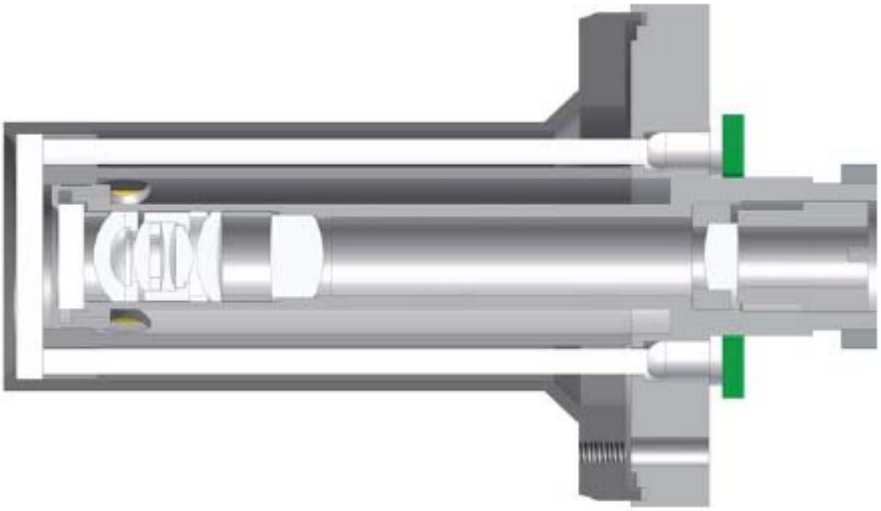

Figure 29 Primary lighting schematic.

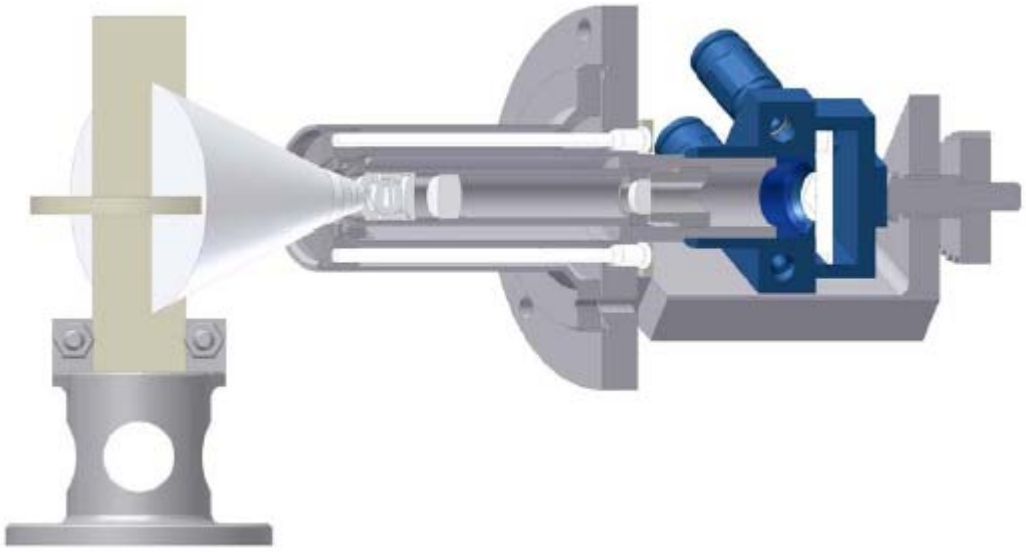

Figure 30 Sample view.

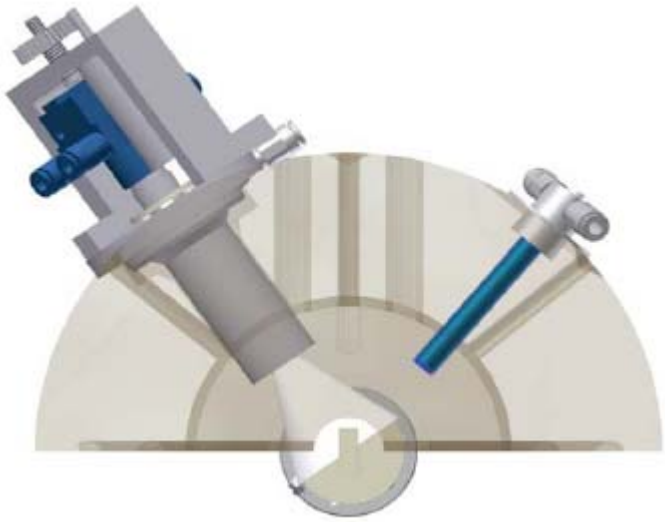

Figure 31 Lighting schematic.

Chapter 3:

Operating the ETC

This chapter describes operating tips for the ETC. For ETC Viewer operation instructions, refer to TRIOS software Online Help.

If the system has been set up according to the instructions in this manual, the rheometer should now be ready to use. However, we recommend that you follow a few extra precautions, described below:

- If you are planning to start your experiment at a high temperature, preheat the system by lowering the head to the measurement gap and allowing upper and lower geometries to rise to the set temperature.
- When you use the cone and plate or parallel plate geometries, it is important to use the correct sample volume. TRIOS software calculates the exact volume required based upon the gap size and geometry diameter. If you know the density of the sample, you can weigh out the correct amount of sample. If you underfill or overfill the gap, you can cause experimental errors in your data.
- When you use the parallel plates, make sure that the oven thermocouple is not touching the plates.
- When you use the parallel plates, if you find that the lower plate is difficult to remove, make sure that you apply a twist to the lower mounting plate—do not apply any force to the ceramic part of the geometry.
- Clean the plates immediately after your experiment with the appropriate solvent. If you are measuring highly viscous materials, or materials that are likely to cure, unscrew the draw rod from the geometry before you raise the head. Stubborn materials can sometimes be removed by heating the plates to a high temperature. The sample will bake and then crumble apart. You can also remove the plates and soak them in an appropriate solvent. It is good practice to always unscrew the draw rod before raising the head. The two plates, together with the sample, can then be removed as a sandwich unit.
- T*orsion rectangular tools only:* You can gently move the thermocouple (inside the oven) closer to the sample to increase performance; however, you should avoid making any sharp bends in the thermocouple sheath. Repeatedly adjusting the positioning may damage the thermocouple and should be avoided.

Operating Hints

Although the response time of the temperature control system is rapid, many of the samples that are of interest at high temperatures (*e.g.*, bitumen, molten polymers, etc.) are very poor conductors of heat. Therefore, the limiting factor in reaching the desired starting temperature is the time it takes for the heat to be conducted into the sample and for the sample to reach thermal equilibrium. You can investigate a sample by carrying out an experiment using no equilibrium time and doing a time sweep experiment (in oscillation mode). If you plot a graph of how the properties of the sample vary with time, you can quickly establish the required equilibrium time.

The tendency of polymers (which are measured while in their molten state) to oxidize can present an additional complication. This problem is generally sample-dependent, but can be reduced by surrounding the sample with an inert atmosphere. To do this, use nitrogen gas rather than air as the feed to the ETC. It also helps if you optimize your test procedures to minimize the amount of time that the sample is held at high temperatures.

Make sure the upper geometry is in place and free to rotate when you perform procedure for mapping of the bearing. For best results, perform the mapping procedure at ambient temperature and without purge gas flowing.

Controlling Cooling

When you set the control valve on the liquid nitrogen unit, you must compromise between the rate of cooling (which is improved by having a large flow rate) and the fineness of control (which is optimized when there is minimal flow rate from the needle valve.) When only a small amount of cooling is required, the solenoid valve is able to open and shut frequently. However, if a large surge of coolant occurred every time the solenoid valve opened, the system temperature would oscillate on either side of the set point.

The setting of the needle valve is affected by the desired set-temperature:

- If cooling is needed at only a few degrees below ambient, then a very small opening is all that is necessary.
- If you operate at -100°C, then a correspondingly higher flow rate of nitrogen is required.

As a general rule, the correct needle valve setting for the desired temperature is one that results in the opening and closing of the solenoid valve for more or less equal periods. Start with a setting of “open two complete turns” and experiment to find the optimum position for your work experiment procedures.

Low Temperature System Maintenance

For maintenance instructions of the cryogenic pressure vessel, please refer to the instructions supplied with the unit. If you purchased the Dewar flask from TA Instruments, the document is titled “Guide to good housekeeping, maintenance and periodic examination of cryogenic pressure vessels.”

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