

AR 2000 Rheometer



Rheometrics Series Operator's Manual

PN 500106.002 Rev. F
Issued May 2004



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.

TA Instruments may have patents, patent applications, trademarks, copyrights, or other intellectual property covering subject matter in this document. Except as expressly provided in written license agreement from TA Instrument, the furnishing of this document does not give you any license to these patents, trademarks, copyrights, or other intellectual property.

TA Instruments Operating Software, as well as Module, Data Analysis, and Utility Software and their associated manuals and online help, are proprietary and copyrighted by TA Instruments. Purchasers are granted a license to use these software programs on the module and controller with which they were purchased. These programs may not be duplicated by the purchaser without the prior written consent of TA Instruments. Each licensed program shall remain the exclusive property of TA Instruments, and no rights or licenses are granted to the purchaser other than as specified above.

Important: TA Instruments Manual Supplement

Please click on the links below to access 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](#)

Table of Contents

Important: TA Instruments Manual Supplement	3
Table of Contents	4
Notes, Cautions, and Warnings	8
Chapter 1: Introducing the AR 2000	9
Overview	9
Warnings	9
Attention	11
Safety and EMC Conformity	13
Specifications	13
Safety	13
EMC	13
La sûreté et EMC Conformité	14
Spécifications	14
Sûreté	14
EMC	14
Lifting and Carrying Instructions	15
Electrical Safety	15
Liquid Nitrogen Safety	16
Handling Liquid Nitrogen	17
If a Person is Burned by Liquid Nitrogen	17
Chemical Safety	17
Usage Instructions	18
Maintenance and Repair	18
Chapter 2: Description of the AR 2000	19
Overview	19
A Brief History of Controlled-Stress Rheometers	19
TA Instruments AR Rheometers	20
Schematics of the AR 2000 Rheometer	20
Instrument Components	21
Chapter 3: Technical Descriptions	23
Overview	23
The Air Bearing	23
Rotational Mapping	24

Auto GapSet Mechanism	25
Zeroing of the Gap	25
Closing the Gap	25
Thermal Compensation	25
Smart Swap™	26
The Peltier Plate	26
Normal Force Transducer	27
Chapter 4: Technical Specifications	29
Overview	29
Specifications	29
Chapter 5: Installation and Operation	33
Overview	33
Removing the Packaging and Preparing for Installation	33
Installation Requirements	34
In	34
On	34
Near	34
Away from	34
Connecting the System Together	35
Connecting the Rheometer to the Electronics Control Box	35
Connecting the Computer to the Electronics Control Box	35
Connecting Air and Water to the Rheometer	36
Using Smart Swap™	37
Installing the Peltier Plate	37
Removing the Peltier Plate	38
Setting Up the Concentric Cylinder System	39
Changing the Cup	40
Using the ETC	41
Installing the Low Temperature Accessory	44
Operating Hints	47
Controlling Cooling	47
Low Temperature System Maintenance	47
General Operating Guidelines	48
Do	48
Do Not	48
Keypad Functionality	49
Levelling the Rheometer	50

Checking Your System	50
Calibrating the Rheometer	51
Shut-Down Procedure	51
Cleaning the Filter Regulator Assembly	52
Chapter 6: Measuring Systems	53
Overview	53
General Description	53
Geometry Materials	53
Stainless Steel	53
Aluminium	53
Plastic	54
Cone and Plate	54
Parallel Plate	55
Concentric Cylinders	56
Using the Stress and Shear Rate Factors	57
Choosing the Best Geometry	58
Cone and Plate/Parallel	58
Plate Systems	58
Angles	58
Diameters	58
Material	59
Preventing Solvent Evaporation	60
Preventing Slippage at Sample/Geometry Interface	60
Removing the Air-Bearing Clamp	61
Attaching a Geometry	62
Ensuring that the Sample is Loaded Correctly	63
Chapter 7: Using the Upper Heated Plate	65
Introduction to the Upper Heated Plate	65
Attaching the Upper Heated Plate to the AR 2000	66
Configurations for the Cooling Water	68
Connecting the Cooling Control Unit	69
Using Circulating Fluids Other Than Water	70
Connecting and Disconnecting the Geometry Holder	72
Connecting the Geometry and Holder	72
Removing the Geometry and Holder	72
Configuring the Upper Heated Plate	73

Calibration of the Upper Heated Plate	74
Clamping the Air Bearing	77
Using an Inert Gas Atmosphere	78
Using the Sample Cover	79
Chapter 8: Do's and Don'ts	81
Overview	81
DO	81
DON'T	82
Appendix A: Useful Information	83
Moments of Inertia	83
Calculations for Moments of Inertia	83
Cone	83
Cylinder	84
Appendix B: Symbols and Units	85
Appendix C: Geometry Form Factors	87
Cone/Plates	87
Concentric Cylinder Dimensions	87
Appendix D: LCD Display Messages	89
Power On Messages	89
Initialising	89
Bearing overspeed	89
Bearing pressure too low	90
Encoder index not found	90
Nf gauge fault	90
Nf temp sensor fault	90
Operator stop event	90
Power cable fault	90
Signal cable fault	90
Temp sys element fault	90
Temp system environment	90
Temp system sensor fault	91
Other Messages	91
Appendix E: TA Instruments ETC Kits	93
ETC Torsion Rectangular Kit (543307.901)	93
ETC Parallel Plate Kit (543306.901)	93
ETC Disp. Parallel Plate Kit (543308.901)	93
Index.....	95

Notes, Cautions, and Warnings

The following conventions are used throughout this guide to point out items of importance to you as you read through the instructions.

A NOTE highlights important information about equipment or procedures.



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



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

Chapter 1

Introducing the AR 2000

Overview

The TA Instruments AR 2000 Rheometer is a controlled stress/controlled rate rheometer capable of handling many different types of samples, using a range of geometry sizes and types.

This manual relates to all hardware aspects of the AR 2000 Rheometer. For complete information on the operation of the instrument, you may also have to refer to the relevant software manuals supplied with the instrument.

Should you find that something in this manual is unclear, or if you feel certain information should be added, please do not hesitate to contact us directly at our Leatherhead, UK office.

TA Instruments Ltd.
Bilton Centre, Cleeve Road
Leatherhead, Surrey. KT22 7UQ

Tel: +44-1372-360363
Fax: +44-1372-360135
Email: info@taeurope.co.uk

Alternatively, please contact your local office directly or speak to your area sales representative.

This chapter describes some important safety information. Please read this information thoroughly before proceeding.

Warnings

Please make sure that you read the following warnings BEFORE using this equipment. This section contains information that is vital to the safe operation of the AR 2000.



WARNING: This equipment must not be mounted on a flammable surface if low flashpoint material is being analyzed.



WARNING: An extraction system may be required if the heating of materials could lead to liberation of hazardous gasses.



WARNING: It is recommended that this instrument be serviced by trained and skilled TA Instruments personnel at least once a year.



WARNING: There may be a danger of explosion if the lithium battery is incorrectly replaced. It should be replaced only with the same type, contact TA Instruments for information. Dispose of used batteries according to the battery manufacturers instructions. If in doubt, contact TA Instruments.



WARNING: The material used on the top surface of the Peltier plate is hard, chrome-plated copper and the material used for the 'skirt' of the Peltier is stainless steel. Therefore, use an appropriate cleaning material when cleaning the Peltier plate.



WARNING: The internal components of the AR 2000 ETC are all constructed from chemically resistant materials, and can therefore be cleaned with standard laboratory solvents. The only exception is the cladding for the thermocouples, which should not be immersed in a solvent for long periods. Use a small amount of solvent on a soft cloth and wipe the soiled area gently. This procedure should never be conducted at any temperature other than ambient.



WARNING: During the installation or reinstallation of the instrument, ensure that the external connecting cables (*i.e.*, data, RS232 etc.) are placed separate from the mains power cables. Also, ensure that the external connecting cables and the mains power cables are placed away from any hot external parts of the instrument.
Note: Ensure that the mains power cable is selected such that it is suitable for the instrument that is being installed or reinstalled, paying particular attention to the current rating of both the cable and the instrument.



WARNING: Before switching the instrument on, apply the air to the instrument and switch on the water supply to the Peltier system (if used).



WARNING: During operation, extreme hot or cold surfaces may be exposed. Take adequate precautions. Wear safety gloves before removing hot or cold geometries.



WARNING: Liquid nitrogen can cause rapid suffocation without warning. Store and use in an area with adequate ventilation. Do not vent liquid nitrogen in confined spaces. Do not enter confined spaces where nitrogen gas may be present unless the area is well ventilated. The warning above applies to the use of liquid nitrogen. Oxygen depletion sensors are sometimes utilized where liquid nitrogen is in use.



WARNING: The various surfaces and pipes of the ETC and the supply Dewar can get cold during use. These cold surfaces cause condensation and, in some cases, frost to build up. This condensation may drip to the floor. Provisions to keep the floor dry should be made. If any moisture does drip to the floor, be sure to clean it up promptly to prevent a slipping hazard.



WARNING: Always unplug the instrument before performing any maintenance.



WARNING: No user serviceable parts are contained in the rheometer. Maintenance and repair must be performed by TA Instruments or other qualified service personnel only.



WARNING: This instrument must be connected to an earthed (grounded) power supply. If this instrument is used with an extension lead, the earth (ground) continuity must be maintained.

Attention



Cet instrument ne doit être en aucun cas installé sur une surface inflammable lors de l'analyse d'échantillons ayant un faible point d'éclair.



Une bouche d'extraction est nécessaire lors de la combustion de matériaux libérant des gaz toxiques.



Il est recommandé que cet appareil soit révisé au moins une fois par an par un ingénieur TA Instruments.



Les piles au lithium de rechange doivent être impérativement du même type que celles d'origine. Dans le cas contraire, un risque d'explosion reste possible. Pour plus d'informations, contacter TA Instruments.
Ne pas jeter de piles au lithium usagées. Celles-ci doivent être recyclées.



La surface supérieure en cuivre de la plaque Peltier est recouverte de chrome. Et la surface latérale est recouverte d'acier. Il est important d'utiliser des produits adéquats, lors du nettoyage du Peltier, qui n'altéreront pas ces deux matériaux.



Les composants internes du four (ETC) monté sur l'AR2000 sont conçus pour résister à toute attaque chimique. Ils peuvent donc être tous nettoyés, à l'aide de solvants quelconques, à l'exception du revêtement des thermocouples, qui ne doivent pas, quant à eux, baigner dans un solvant pendant une longue période. Ceux-ci doivent être nettoyés à température ambiante en frottant légèrement avec un chiffon imbibé de solvant.



Les câbles externes doivent être toujours séparés du câble d'alimentation. S'en assurer à chaque installation. De même, tout câble doit être éloigné de toute source de chaleur (Peltier...).



Avant toute mise en marche, s'assurer que l'arrivée d'eau pour le Peltier (si utilisé) ainsi que l'arrivée d'air pour le moteur sont connectées et que l'eau et l'air circulent.



Les différentes surfaces, tuyaux de l'ETC ainsi que le réservoir d'azote liquide peuvent être exposés à de très basses températures pendant l'utilisation. Ces surfaces froides provoquent de la condensation et peuvent même être à l'origine d'une formation de glace. Cette condensation risque de goutter par terre. Afin d'éviter tout accident dû à un sol glissant, il serait préférable de garder le sol aussi sec que possible.



Lors de toute maintenance, couper l'alimentation.



Toute maintenance ou réparation doivent être effectuées par TA Instruments ou un personnel de service qualifié.



Cet appareil doit être connecté à la terre. Toute rallonge utilisée avec cet appareil doit comporter une masse de sécurité.



Utiliser l'azote liquide avec précautions car une utilisation inadéquate peut provoquer des suffocations. Stocker et utiliser dans une pièce suffisamment ventilée. Ne pas pénétrer dans une pièce remplie d'azote avant d'en avoir évacué le gaz.

Safety and EMC Conformity Specifications

In order to comply with the European Council Directives, 73/23/EEC (LVD) and 89/336/EEC (EMC Directive), as amended by 93/68/EEC; the AR 2000 has been tested to the following specifications:

Safety

This equipment has been designed to comply with the following standards on safety:

- EN 61010-1:1993
Safety requirements for electrical equipment for measurement, control and laboratory use.
EN 61010-1 Amendment 1, 1995
EN 61010-1 Amendment 2, 1995
- EN 6101-2-010: 1994
Particular requirements for laboratory equipment for the heating of materials.
EN 61010-2-010 Amendment 1, 1996
- UL3101-1 First Edition 1993
IEC 1010-2-010: 1992
- CAN/CSA-C22.2 No.1010-1: 1992
IEC 1010-2-010: 1992

EMC

- EN61326-1: 1997
Electrical equipment for measurement, control and laboratory use.
Incorporating:

EN55011: 1998	Conducted Class B
EN55011: 1998	Radiated Class A
EN6100-3-2: 1995	Harmonic current
EN6100-3-3: 1995	Voltage flicker
EN6100-4-2: 1995	ESD
EN6100-4-3: 1996	Radiated RF
EN6100-4-4: 1995	Fast Transient/Burst
EN6100-4-5: 1995	Surge
EN6100-4-6: 1996	Conducted disturbances
EN6100-4-11: 1994	Voltage dips
- AZ/NZS 2064: 1997

La sûreté et EMC Conformité

Spécifications

Afin de se conformer aux directives du Conseil européen, 73/23/EEC (LVD) et 89/336/EEC (directive d'cEmc), comme modifié par 93/68/EEC; l'Ar 2000 a été testé selon les caractéristiques suivantes:

Sûreté

Ce matériel a été conçu pour être conforme aux normes de sécurité suivantes:

- EN 61010-1:1993
Conditions de sécurité pour l'appareillage de mesures électrique, la commande et l'usage de laboratoire.
EN61010-1 Amendment 1, 1995
EN61010-1 Amendment 2, 1995
- EN6101-2-010: 1994
Conditions particulières pour le matériel de laboratoire destine au chauffage des matériaux.
EN61010-2-010 Amendment 1, 1996
- UL3101-1 First Edition 1993
IEC 1010-2-010: 1992
- CAN/CSA-C22.2 No.1010-1: 1992
IEC 1010-2-010: 1992

EMC

- EN61326-1: 1997
Conditions de sécurité pour l'appareillage de mesures électrique, la commande et l'usage de laboratoire.
incorporation
EN55011: 1998 Conducted Class B
EN55011: 1998 Radiated Class A
EN6100-3-2: 1995 Harmonic current
EN6100-3-3: 1995 Voltage flicker
EN6100-4-2: 1995 ESD
EN6100-4-3: 1996 Radiated RF
EN6100-4-4: 1995 Fast Transient/Burst
EN6100-4-5: 1995 Surge
EN6100-4-6: 1996 Conducted disturbances
EN6100-4-11: 1994 Voltage dips
- AZ/NZS 2064: 1997

Lifting and Carrying Instructions

Please follow these recommendations when you move or lift the instrument and its accessories:

- Before moving the rheometer, you should remove any temperature attachments from the SmartSwap™ holder. See Chapter 5 for more information.
- When moving the rheometer, the air-bearing clamp should always be in place, ensuring that the bearing cannot be moved. See Chapter 5 for information on the air-bearing clamp and how it is attached.
- Use two hands to lift the instrument, keeping your back straight as you lift, to avoid possible strain on your back. You should always use two people to lift the instrument.
- Treat the AR 2000 with the same degree of care you would take with any scientific laboratory instrument.

Electrical Safety

Always unplug the instrument before performing any maintenance.

Supply Voltage	110 - 240 Vac
Fuse type	2 x F10 A H250v
Mains Frequency	45 to 65 Hz
Power	1000 watts



WARNING: Because of the high voltages in this instrument, maintenance and repair of internal parts must be performed by TA Instruments or other qualified service personnel only.



Cet instrument étant sous hautes tensions, l'entretien et la réparation des pièces internes doivent être effectués exclusivement par TA instruments ou tout autre personnel de service qualifié.

Liquid Nitrogen Safety



Potential Asphyxiant

WARNING: Liquid nitrogen can cause rapid suffocation without warning. Store and use in an area with adequate ventilation. Do not vent liquid nitrogen in confined spaces. Do not enter confined spaces where nitrogen gas may be present unless the area is well ventilated. The warning above applies to the use of liquid nitrogen. Oxygen depletion sensors are sometimes utilized where liquid nitrogen is in use.



Potentiel Agent asphxyiant

L'azote liquide peut causer des suffocations rapides. Stocker et utiliser dans une zone dotée d'une ventilation adéquate. Ne pas ventiler d'azote liquide dans des espaces confinés. Ne pas pénétrer dans des espaces confinés où le gaz d'azote peut être présent à moins de bien aérer la zone. L'avertissement ci-dessus s'applique à l'utilisation de l'azote liquide. Des capteurs d'épuisement d'oxygène sont parfois utilisés.



Extremes of temperature

During operation, extreme hot or cold surfaces may be exposed. Take adequate precautions. Wear safety gloves before removing hot or cold geometries.



Températures extremes.

Lors du fonctionnement, des surfaces extrêmement chaudes ou froides peuvent être exposées. Prendre toutes précautions nécessaires telles que l'utilisation de gants de protection avant d'enlever les géométries chaudes ou froides.

Handling Liquid Nitrogen

The ETC uses the cryogenic (low-temperature) agent, liquid nitrogen, for cooling. Because of its low temperature [-195°C (-319°F)], liquid nitrogen will burn the skin. When you work with liquid nitrogen, use the following precautions:

Liquid nitrogen evaporates rapidly at room temperature. Be certain that areas where liquid nitrogen is used are well ventilated to prevent displacement of oxygen in the air.

1. Wear goggles or a face shield, gloves large enough to be removed easily, and a rubber apron. For extra protection, wear high-topped, sturdy shoes, and leave your trouser legs outside the tops.
2. Transfer the liquid slowly to prevent thermal shock to the equipment. Use containers that have satisfactory low-temperature properties. Ensure that closed containers have vents to relieve pressure.
3. The purity of liquid nitrogen decreases as the nitrogen evaporates. If much of the liquid in a container has evaporated, analyze the remaining liquid before using it for any purpose where high oxygen content could be dangerous.

The oven inner doors have a trough around the bottom of the element assembly for collection of excess liquid nitrogen. Any excess fluid collected will drain out from the oven at the lower outer edge.

If a Person is Burned by Liquid Nitrogen

1. IMMEDIATELY flood the area (skin or eyes) with large quantities of cool water, then apply cold compresses.
2. If the skin is blistered or if there is a chance of eye infection, take the person to a doctor IMMEDIATELY.

Chemical Safety

Do not use hydrogen or any other explosive gas with the ETC.

Use of chlorine gas will damage the instrument.

If you are using samples that may emit harmful gases, vent the gases by placing the instrument near an exhaust.

Usage Instructions

Before connecting the rheometer to auxiliary equipment, you must ensure that you have read the relevant installation information. Safety of the rheometer may be impaired if the instrument:

- Shows visible damage
- Fails to perform the intended measurements
- Has been badly stored
- Has been flooded with water
- Has been subjected to severe transport stresses.

Maintenance and Repair



CAUTION: Adjustment, replacement of parts, maintenance and repair should be carried out by trained and skilled TA personnel only. The instrument should be disconnected from the mains before removal of the cover.

Le réglage, le remplacement des pièces, l'entretien et la réparation devraient être effectués exclusivement par le personnel qualifié de TA Instruments. Avant l'ouverture du châssis, débrancher l'instrument.



WARNING: The cover should only be removed by authorized personnel. Once the cover has been removed, live parts are accessible. Both live and neutral supplies are fused and therefore a failure of a single fuse could still leave some parts live. The instrument contains capacitors that may remain charged even after being disconnected from the supply.

Le châssis doit être retiré exclusivement par le personnel autorisé. Une fois le châssis retiré, les pièces connectées à l'alimentation sont accessibles. L'instrument contient plusieurs fusibles. L'instrument contient des condensateurs qui peuvent rester chargés même après avoir été débranchés.

Chapter 2

Description of the AR 2000

Overview

This chapter describes the main components of the rheometer and provides technical information on performance and design. Please read this chapter thoroughly to become familiar with the nomenclature used throughout this manual.

A Brief History of Controlled-Stress Rheometers

Sir Isaac Newton (c.1700) was the first to formulate a mathematical description of a fluid's resistance to deform or flow when a stress was applied to it. He described this resistance as the *viscosity*. It is mathematically described as the shear stress divided by the shear rate or strain. Until Couette developed the first rotational viscometer (c.1890), viscosity was measured using stress driven (gravity) flow. Many of today's techniques still use this principle, such as flow cups, U-tubes, capillaries, etc.

The development of an electromechanical instrument, using synchronous motors, and the electronic versions, using controlled speed servomotors, made controlled rate the widely used technique for versatile rheological instruments for many years.

The first controlled-stress instrument, capable of continuous rotation, was developed by Davis, Deer, and Warburton (1968 *J.Sci. Instr.* 2, I, 933-6) at the London School of Pharmacy. This instrument used an air turbine and an air bearing. In the early 1970's, a second generation of instruments was developed, using an induction motor drive to avoid the problems associated with the air turbine. These, however, were restricted to a maximum torque of 5000 μNm .

TA Instruments AR Rheometers

The TA Instruments AR Rheometers are fifth-generation instruments that function as either controlled-stress or controlled-rate instruments.

The rheometers are designed to fulfill the requirements of measurement as implied by the full meaning of the term *rheology*—defined as the "study of the deformation and flow of matter."

Deformation is measured in the nondestructive region of elastic or viscoelastic deformation. This can give invaluable information concerning the microscopic interactions in the test material, as well as measuring the shear stress/shear rate relationships at higher stresses.

In the controlled-stress technique, the stress can be applied and released at will, and the actual behavior of the sample can be measured directly. This is not usually possible with conventional controlled-shear rate instruments. In addition, most real-life situations can be simulated more accurately using controlled-stress measurements.

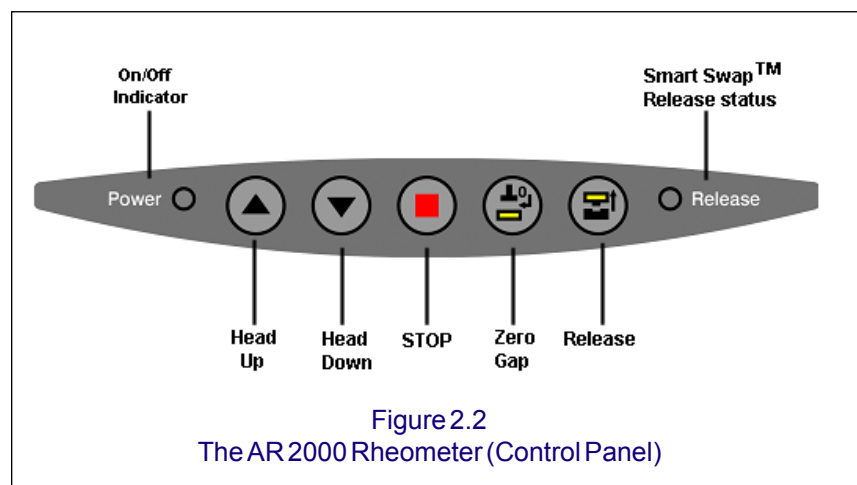
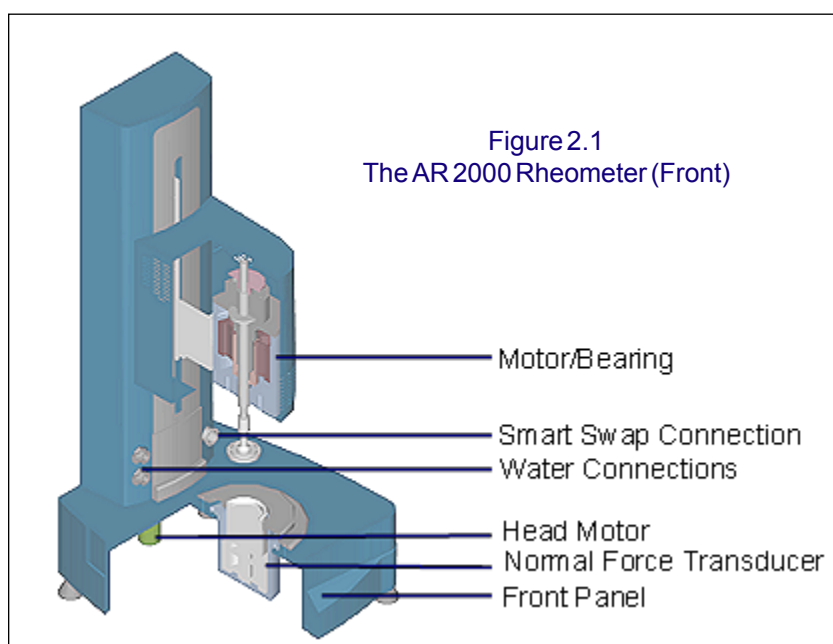
Schematics of the AR 2000 Rheometer

The parts of the AR 2000 are shown in the figures in this section as follows:

Figure 2.1 shows a schematic of the front of the rheometer.

Figure 2.2, shows the control panel.

The rear of the instrument is shown in Figure 2.3 on the next page.



Instrument Components

The instrument consists of a main unit mounted on a cast metal stand, with the electronic control circuitry contained within a separate electronics control box. Figure 2.4 shows the rear of the electronics control box.

The AR 2000 Rheometer contains an electronically-controlled induction motor with an air bearing support for all the rotating parts. The drive motor has a hollow spindle with a detachable draw rod inserted through it. The draw rod has a screw-threaded section at the bottom, which allows the geometry to be securely attached.

The measurement of angular displacement is done by an optical encoder device. This can detect very small movements down to 40 nRad. The encoder consists of a non-contacting light source and photocell arranged either side of a transparent disc attached to the drive shaft. On the edge of this disc are extremely fine, accurate photographically-etched radial lines. Therefore, this is a diffraction grating. There is also a stationary segment of a similar disc between the light source and encoder disc. The interaction of these two discs results in diffraction patterns that are detected by the photocell.

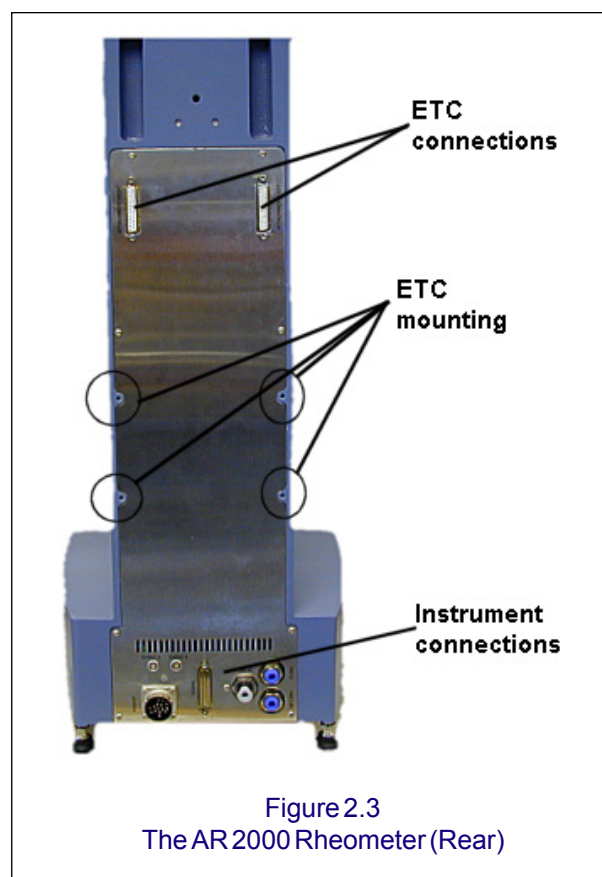


Figure 2.3
The AR 2000 Rheometer (Rear)

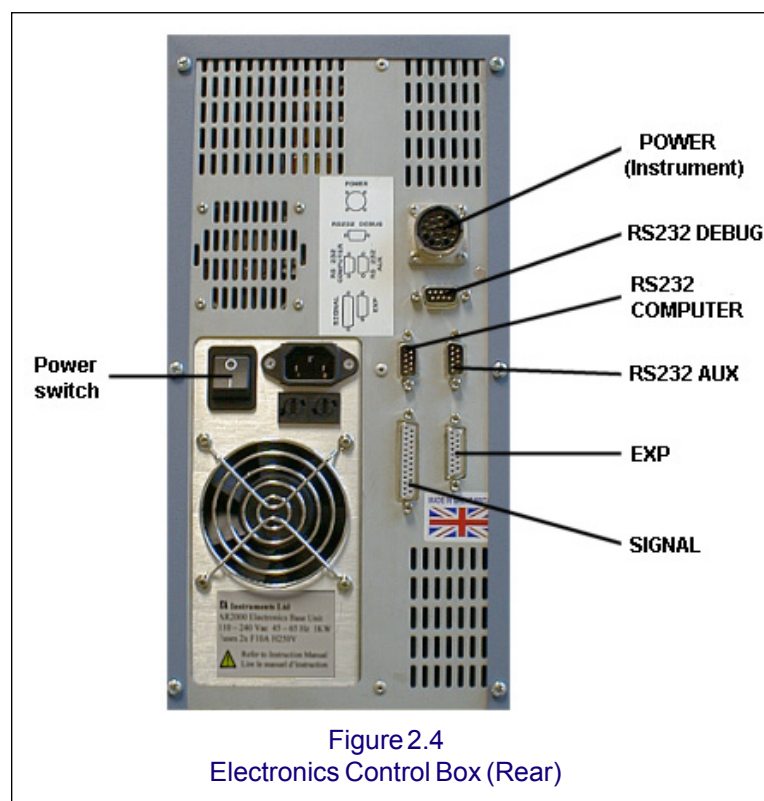


Figure 2.4
Electronics Control Box (Rear)

As the encoder disc moves when the sample strains under stress, these patterns change. The associated circuitry interpolates and digitizes the resulting signal to produce digital data. This data is directly related to the angular deflection of the disc, and, therefore, the strain of the sample.

The main electronics are housed in a separate control box. (The interplay between the rheometer/electronics and controller are explained in more detail in Chapter 5.)

Temperature control is achieved via interchangeable temperature options. These are discussed in more detail in Chapter 3.

Chapter 3

Technical Descriptions

Overview

In order to fully utilize the advanced capabilities available with the AR 2000 Rheometer, some of the important components require a more detailed explanation. This chapter describes in detail the design and functions of the:

- Air bearing
- Auto gap set device
- Smart Swap™
- Peltier plate
- Normal force transducer.

The Air Bearing

As its name suggests, an air bearing uses air as the lubricating medium. This allows virtually friction-free application of torque.

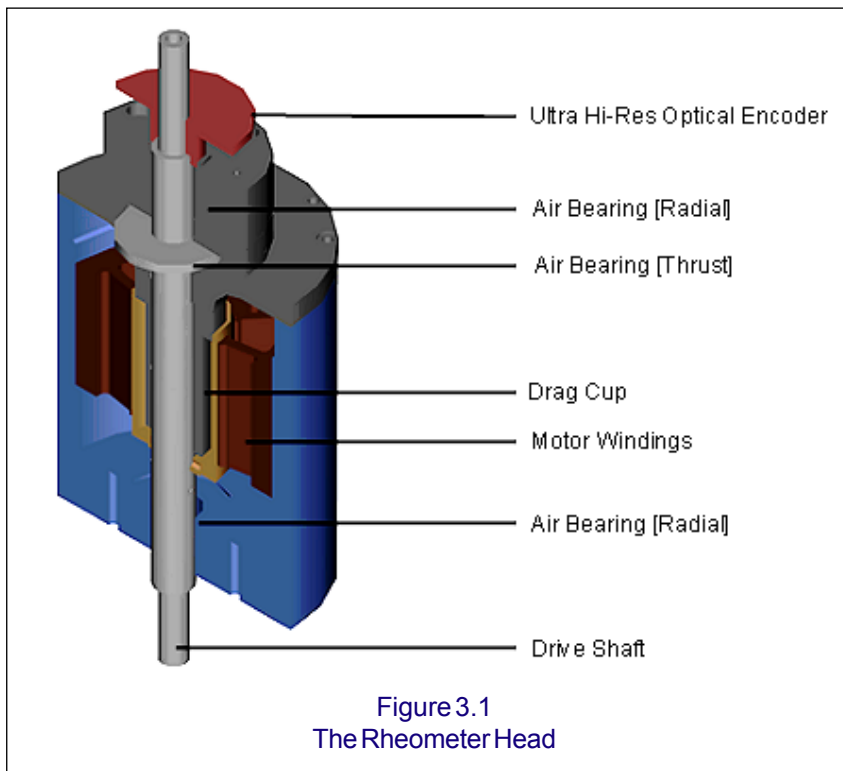
The design of an air bearing is a compromise between several characteristics such as air consumption, friction, stiffness, and tolerance to contamination and misuse.

The amount of air consumed is related to the pressurized bearing clearance. To minimize air consumption, a small clearance ($<10\mu\text{m}$) is needed. However, as air has a finite viscosity (0.0018 mPa.s), small gaps give rise to high shear rates and, correspondingly, the friction increases.

If large gaps are used, the shear rate is lowered and friction is reduced, but the stiffness of the air bearing is also reduced.

Thus, a compromise in the design of an air bearing is needed for optimal performance.

The air bearing used in the AR 2000 Rheometer uses a mixture of proven bearing techniques with novel materials. The surfaces can be easily machined to tolerances of less than $1\mu\text{m}$, providing an extremely smooth finish.



A schematic of the Air Bearing and the other main components of the rheometer head is shown in Figure 3.1 above.

The bearing is designed to be virtually friction- free, so that it moves under the smallest of forces. Even extremely small manufacturing variations in the bearing can be sufficient to make it rotate. Therefore, to ensure that the bearing rotation is steady throughout a full 360°, a process called Rotational Mapping, which is explained in the next section, is carried out.

Rotational Mapping

As explained previously, any real air bearing will have small variations in behavior around one revolution of the shaft.

By combining the absolute angular position data from the optical encoder with microprocessor control of the motor, these small variations can be mapped automatically and stored, since the variations are consistent over time, unless changes occur in the air bearing.

The microprocessor can allow for these automatically by carrying out a baseline correction of the torque. This results in a very wide bearing operating range, without operator intervention; *i.e.*, a confidence check in bearing performance.

Instructions for performing the rotational mapping can be found in the Rheology Advantage™ online help.

Auto GapSet Mechanism

The auto gap set facility has three major functions, as follows:

- Automatic setting of gaps via software
- Programmed gap closure
- Thermal gap compensation.

These features are described in more detail on the following pages.

Zeroing of the Gap

It is important that you use a reproducible gap zeroing technique to reduce errors from such factors as operator-to-operator techniques. The automation of gap zeroing on the rheometer minimizes these errors.

Closing the Gap

Once you have set the gap and loaded the sample, the head is lowered. The velocity and deceleration of the head as it is lowered is controlled via the 'automatic gap options' set in the Rheology Advantage software. There are four closure options available with the AR 2000 Rheometer—Standard, Linear, Exponential, and Normal Force. The options available are described in detail in the online help for the rheology software.



CAUTION: Keep hands and fingers away from the plate during head movement.

S'assurer que les mains ou doigts ne soient pas entre le peltier et la géométrie lors du mouvement de la tête de l'instrument.

Thermal Compensation

When a wide temperature range is used for an experiment, the metallic rheometer parts and the measurement geometries can heat or cool causing expansion or contraction of the measurement system gap. A typical expansion value for stainless steel geometries is $0.5 \mu\text{m}^{\circ}\text{C}^{-1}$. The auto gap-set facility compensates for these changes. Therefore, regardless of temperature, you can be confident that the gap remains constant.

Smart Swap™

The AR2000 features "Smart Swap" technology that automatically senses the temperature control system present and configures the rheometer operating software accordingly, loading all relevant calibration data. The use of this feature is covered later in Chapter 5.

The Peltier Plate

Temperature control in the standard configuration is via a Peltier system (both a plate and concentric cylinder system are available), which uses the Peltier effect to rapidly and accurately control heating and cooling. The Peltier system uses a thermoelectric effect. This functions as a heat pump system with no moving parts, and is ideally suited to rheological measurements. By controlling the magnitude and direction of electric current, the Peltier system can provide any desired level of active heating or cooling directly in the plate. A schematic of the Peltier plate is shown in Figure 3.2.

Since the Peltier system operates as a heat pump, it is necessary to have a heat sink available. The heat sink removes unwanted waste heat from the plate. This heat sink is normally in the form of a reservoir, containing a few liters of water, plus a small pump that can provide sufficient flow rate through the Peltier heat exchanger jacket built into the plate.

The reservoir fluid will become warm with the prolonged use of the Peltier at high or sub-ambient temperatures.

If your temperature range is 20°C below and 60°C above ambient, the water bath should be at room temperature. If, however, you wish to work at lower or higher temperatures, the water bath temperature needs to be altered accordingly (*i.e.*, the Peltier system will work most efficiently at a temperature range that is 15°C above and below the water bath temperature.). When routinely using the Peltier system at temperatures above 100°C, it is recommended that you connect the system to a main water supply.

The flow rate through the Peltier does not need to be high. A flow rate of at least 0.5 litre min⁻¹ is adequate. When working at the Peltier's lowest temperature range, increasing the flow rate to >1 liter min⁻¹ will give a better performance. If this flow rate is not maintained, the temperature control system will lose control and the system will only heat.

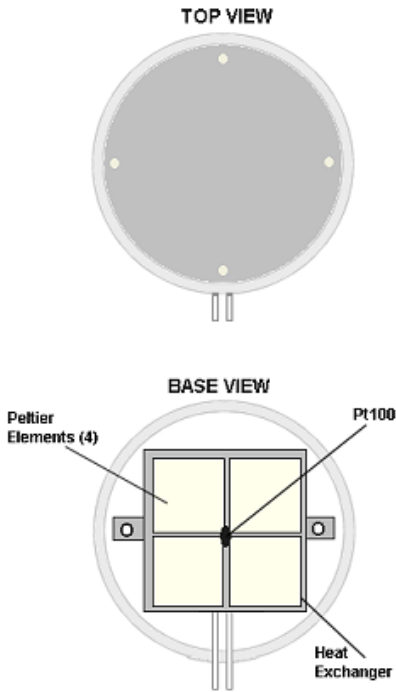


Figure 3.2
The Peltier Plate

Peltier	Temperature Range
tank & pump	-5°C to 100°C
pumped water supply (20°C)	-20°C to 200°C
water at 60°C	10°C to 200°C
water at 40°C	0°C to 200°C
water at 1°C	-30°C to 180°C
fluid at -20°C	-40°C to 160°C



CAUTION: The Peltier Plate may be damaged by operating the instrument without a flow of water through the Peltier system. There is a Peltier Overheat protection device that will activate if the device becomes too hot.

Sans écoulement d'eau, le système Peltier peut être endommagé. Un dispositif de protection a été conçu pour se déclencher en cas de surchauffe.

Normal Force Transducer

When a viscoelastic liquid is sheared, a force can be generated along the axis of rotation of a cone or parallel plate geometry. For this to happen, the structure responsible for the elasticity must not be completely disrupted by steady shear.

For this reason, colloids, suspensions, etc., although elastic at rest, become effectively inelastic under steady shear and can show negative normal forces due to inertial effects. However, polymer solutions and melts, and products incorporating them, are typically elastic under shear because of the long lifetime of the molecular entanglement.

Normal force measurements are made with cone and plate or parallel plate geometries; therefore, it is important to use a method to detect the force that does not allow significant changes in the gap. This would result in the actual shear rate varying with normal force, due to deflections of the force-detecting component.

The AR 2000 Rheometer keeps the upper geometry positioned as accurately as is possible with an air bearing, and movement is kept to an absolute minimum. This ensures good bearing performance.

The force is detected on the static lower measuring geometry assembly using high sensitivity load cell technology. This results in a fast response, wide range signal, which is easy to calibrate, and has a genuine normal force measurement capability.



CAUTION: During sample loading and measurement, the normal force transducer is protected from overload. However, take care when cleaning or attaching accessories to the lower plate that you do not exceed the maximum normal force.

Le capteur de force normale est protégé contre toute surcharge. Cependant, prendre soin de ne pas dépasser la force normale maximale lors de toute manipulation (nettoyage, changement de plaque...).

Chapter 4

Technical Specifications

Overview

This chapter contains the technical specifications for the AR 2000 Rheometer. You can obtain further information from your local Sales Representative.

Specifications

The following specifications apply to the TA Instruments AR 2000 Rheometer:

Table 4.1
AR 2000 Rheometer Dimensions

Accessory (Electronics Base)		
Width	7.25 in.	(18.5 cm)
Height	14.75 in.	(37.5 cm)
Depth	17.75 in.	(45 cm)
Weight	38.1 lbs	(17.3 kg)

Module (Instrument Base)		
Width	11.75 in.	(30 cm)
Height	26.5 in.	(67 cm)
Depth	12.5 in.	(32 cm)
Weight	62.2 lbs	(28.7 kg)

Table 4.2
AR 2000 Rheometer Specifications

Supply Voltage	110 – 240 Vac
Supply Frequency	45 to 65 Hz
Power	1000 Watts
Torque Range	0.1 μ Nm to 200 mNm
Frequency Range	0.12 μ Hz to 100 Hz
Angular Velocity Range	Controlled Stress: 10 ⁻⁸ to 300 Rad s ⁻¹ Controlled Strain: 10 ⁻² to 300 Rad s ⁻¹
(table continued)	

Table 4.2
AR 2000 Rheometer Specifications

Angular Displacement Resolution	40 nRad
Minimum Strain	0.00006
Normal Force Range	1 g to 5000 g

Table 4.3
Peltier Plate System Specifications

Temperature Range tank & pump pumped water supply (20°C) water at 60°C water at 40°C water at 1°C fluid at -20°C	 -5°C to 100°C -20°C to 200°C 10°C to 200°C 0°C to 200°C -30°C to 180°C -40°C to 160°C
Typical Ramp Rate Ramp Rate (20 to 100 °C) (100 to 150 °C)	 30 °C min ⁻¹ 50 °C min ⁻¹ 25 °C min ⁻¹
Pt100 Internal Resolution	0.01 °C

Table 4.4
Optional Accessory Specifications for
Environmental Test Chamber Module (ETC)

Temperature Range No cooling LN2 cooling	 50°C to 600°C -150°C to 600°C
Typical Ramp Rate	maximum ramp rate 25°C/min
Internal Resolution	0.02 °C

Table 4.5
Optional Accessory Specifications for
Peltier Concentric Cylinder System

Temperature range with tank and pump with plumbed water supply with fluid at -20°C	0°C to 100°C -10°C to 150°C -40°C to 100°C
Ramp Rate Cooling Heating	15°C/min maximum 13°C/min maximum
Pt100 Internal Resolution	0.01 °C

Table 4.6
Optional Accessory Specifications for Upper Heated Plate

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

Chapter 5

Installation and Operation

Overview

Normally the installation of your new system will be carried out by a member of the TA Instruments sales or service staff, or their appointed agents, and it will be ready for you to use. However, should you need to install or relocate the instrument, this chapter provides the necessary instructions.

Removing the Packaging and Preparing for Installation

If needed, the first step is to carefully remove all items from any and all packaging. We recommend that you retain all packaging materials in case the instrument has to be shipped back to TA Instruments at some point in the future (for example, in the case of some upgrades).

Please follow these recommendations when you move or lift the instrument and its accessories:

- Always remove the temperature control module from the rheometer before attempting to move it. Details on how to do this can be found later in this section (SmartSwap™).
- When moving the rheometer, the air-bearing clamp should always be in place, ensuring that the bearing cannot be moved.

1. Insert the draw rod into the top of the rheometer.

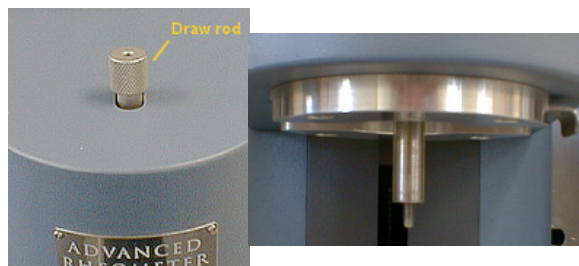


Figure 5.1
Inserting the Draw Rod

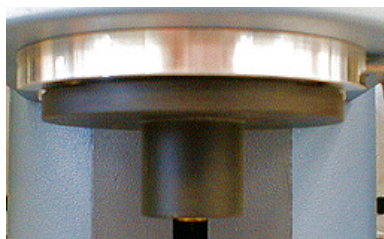


Figure 5.3
Performing Step 3

2. Next, slide the bearing lock into place (you may need to turn the shaft so that the flats line up with the lock.)

3. Push the air-bearing clamp up onto the draw rod. Hold it in place while turning the knob at the top in a clockwise direction.



CAUTION: Always hold the clamp and turn the knob - never the other way round.

Toujours tenir la géométrie et tourner la molette - jamais le contraire.



Figure 5.2
Installing the Bearing Lock

- Treat the AR 2000 Rheometer with the same degree of care you would take with any scientific laboratory instrument.

If your instrument does not have a bearing lock, ignore this step. This mechanical lock has been removed from current production units as the software Bearing Lock, implemented with the Mobius Drive, provides the same function without the risk of leaving it in place at the start of a measurement.

Installation Requirements

It is important to select a location for the instrument using the following guidelines.

Choose a location that is...

In

- A temperature-controlled area. ($22^{\circ}\text{C} \pm 4^{\circ}\text{C}$, relative humidity $50 \pm 10\%$).
- A clean environment (indoor use).
- An area with ample working and ventilation space around the instrument, approximately 2 meters in length, with sufficient depth for a computer and its keyboard.

On

- A stable, vibration-free work surface.

Near

- A power outlet. (Mains supply voltage fluctuations not to exceed $\pm 10\%$ of the nominal voltage, installation category 2.)
- Your computer.
- Sources of compressed lab air and purge gas supply for use during cooling and sub-ambient experiments. A compressed air supply that is capable of supplying clean, dry, oil free air at an approximate pressure of 30 psi (~ 2 Bar) at a flow rate of 50 liters⁻¹. The dew point of the air supply should be -20°C or better.

Away from

- Dusty environment (pollution degree 1).
- Exposure to direct sunlight.
- Poorly ventilated areas.

After you have decided on the location for your instrument, refer to the following sections to unpack and install the AR 2000 Rheometer.

NOTE: Internal Fuse: FS1 & FS2 on cmd 069 pcb. It is strongly recommended that the internal fuse be replaced only by trained and skilled TA Instrument personnel.

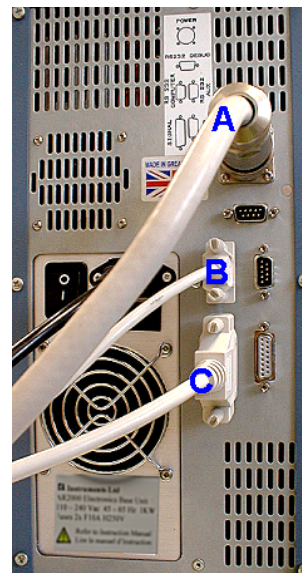
Connecting the System Together

Connecting the system together should present no problems, as long as you use instructions found in the following sections.

Connecting the Rheometer to the Electronics Control Box

The Electronics Control Box forms the link between the rheometer and the computer. All the required processing is done within the control box. The following steps should be followed to connect the two units together (refer to Figure 5.4).

1. Push the female end of the Power cable into the Power port on the back of the rheometer and the other end in the Power port on the back of the control box (Cable A).
2. Push the D-type cable into the Signal port on the back of the rheometer and connect the other end to the Signal port on the back of the control box (Cable C).



Connecting the Computer to the Electronics Control Box

The electronics control box and computer are connected via a single RS232 cable, which is supplied with the system. One end of the cable has a 9-pin female connector; the other end has a 9-pin male connector.

1. Push the 9-pin female connector into the 9-pin socket marked 'Computer' on the back plate of the controller (Cable B, Figure 5.4).
2. Push the 9-pin male connector into the serial port socket on the back of the socket on the computer.

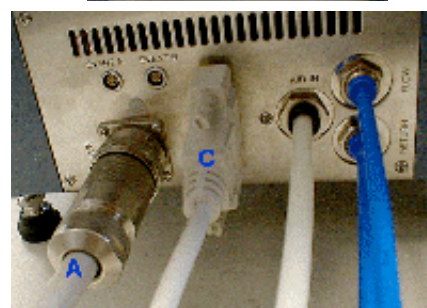


Figure 5.4
Cable Connections

NOTE: You must configure the software for the appropriate communications port—refer to the online help for instructions on how to do this.

Vous devez configurer le logiciel en fonction du port de transmissions utilisé—se référer à l'aide fournie dans le logiciel.

Connecting Air and Water to the Rheometer

Refer to Figure 5.4 on the previous page for information on the location of the relevant connections in the instructions below.

1. Connect a supply of cooling water the flow and return connections at the rear of the rheometer
2. Connect the air supply (from the air regulator assembly) to the '**air in**' connection.

Using Smart Swap™

The following sections explain how to attach/detach temperature modules using Smart Swap™. Note, however, that the installation and removal procedures are essentially the same for all modules.

The following modules are covered:

- Peltier plate
- Concentric cylinders
- Environmental Test Chamber (ETC).

Installing the Peltier Plate

1. Press the '**Release button**' on the control panel as seen in Figure 5.5. A continuous green light indicates that the attachment can be fitted.

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

Le déverrouillage restera seulement actif pendant 10 secondes.

2. Fit the attachment as shown in Figure 5.6 below, ensuring it is aligned correctly.

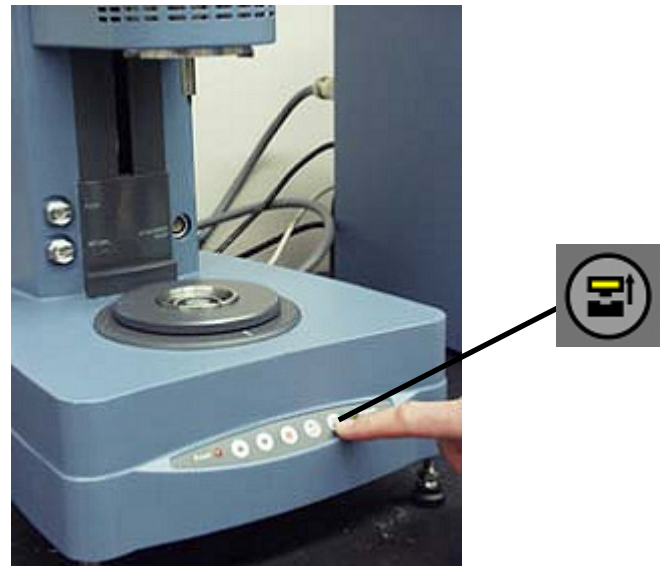


Figure 5.5
Press the Release Button

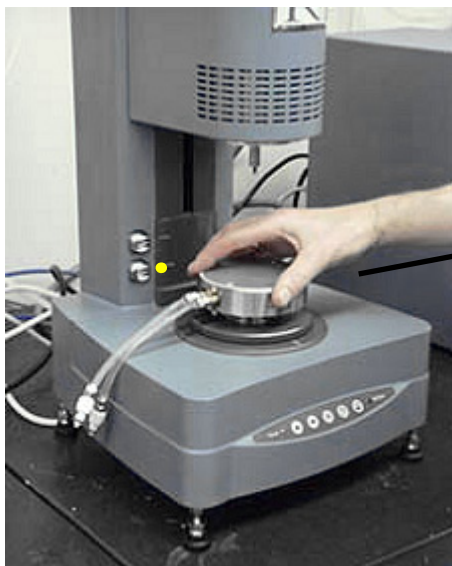


Figure 5.6
Fitting the Attachment



Figure 5.7
Connecting Power and Fluid Cables

3. Connect the power and fluid cables. See Figure 5.7.
4. When the green status light goes out, the rheometer is ready for use.

Removing the Peltier Plate

1. Press the '**Release button**' on the control panel (see Figure 5.8). A flashing green light indicates that the attachment can be unplugged.
2. Press the Release button again. A continuous green light indicates that you can remove the attachment.
3. Remove the attachment from the rheometer. See Figure 5.9.

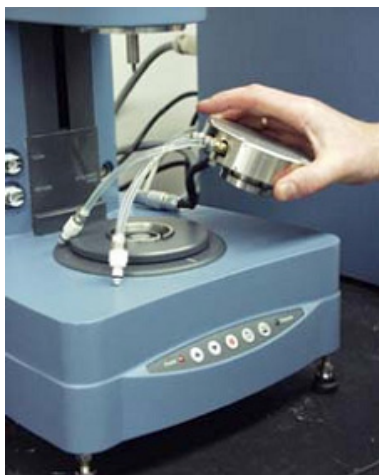


Figure 5.9
Removing the Peltier Plate

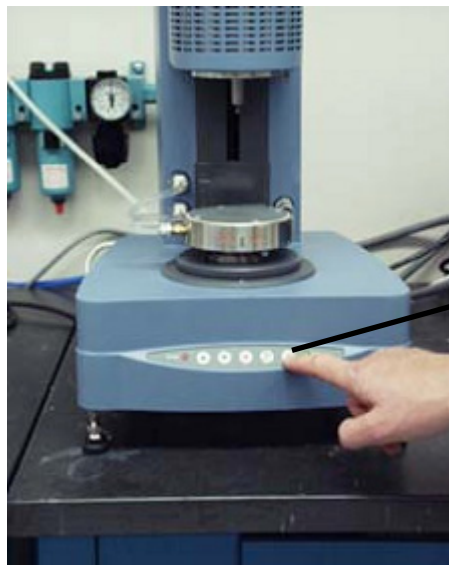


Figure 5.8
Releasing the Attachment

NOTE: The release state will stay active for 10 seconds and then revert to locked.

Le déverrouillage restera actif pendant 10 secondes. A l'issue de ces 10 secondes la plaque sera verrouillée automatiquement.

Setting Up the Concentric Cylinder System

The concentric cylinder system consists of a water jacket, an inner cylinder (the cup) and a rotor (or bob).

To set up the concentric cylinder system, follow these steps:

1. Raise the rheometer head to the top most position.
2. Press the '**Release button**' on the control panel as seen in Figure 5.10. A continuous green light indicates that the attachment can be fitted.

NOTE: The release state will only stay active for 10 seconds.
Le déverrouillage restera actif pendant 10 secondes.

3. Fit the cylinder attachment, ensuring it is aligned correctly.

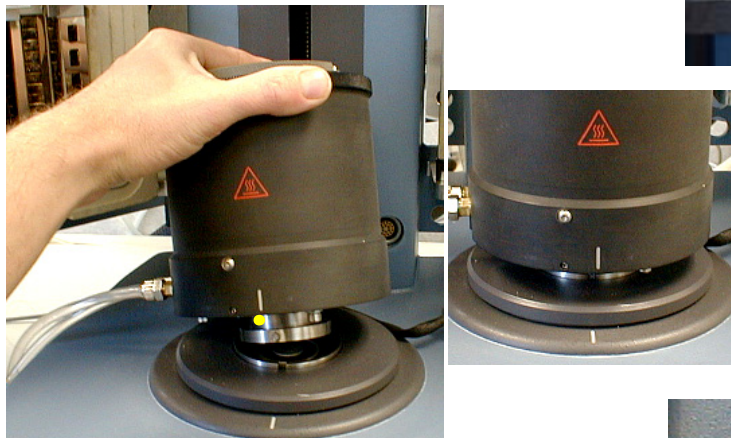


Figure 5.11
Fitting the Cylinder Attachment

4. Connect the power and fluid cables as shown in Figure 5.12 to the right.
5. When the green status light goes out, the lower cup is correctly installed.
6. Lift the rheometer head and attach the correct rotor (bob) to the air bearing.

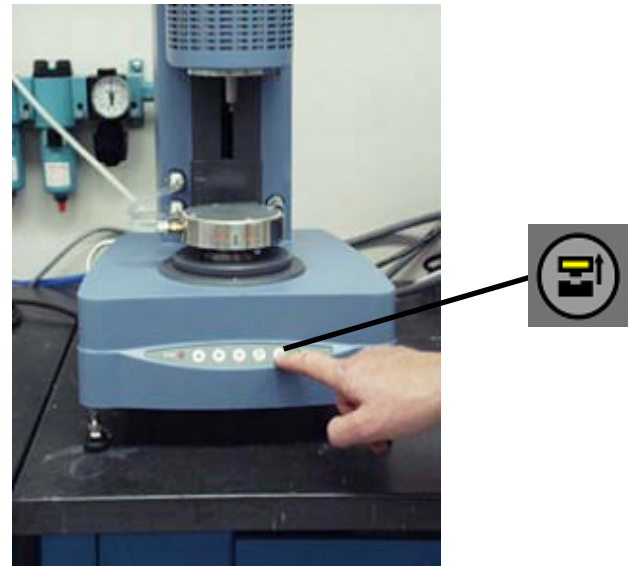


Figure 5.10
Releasing the Attachment

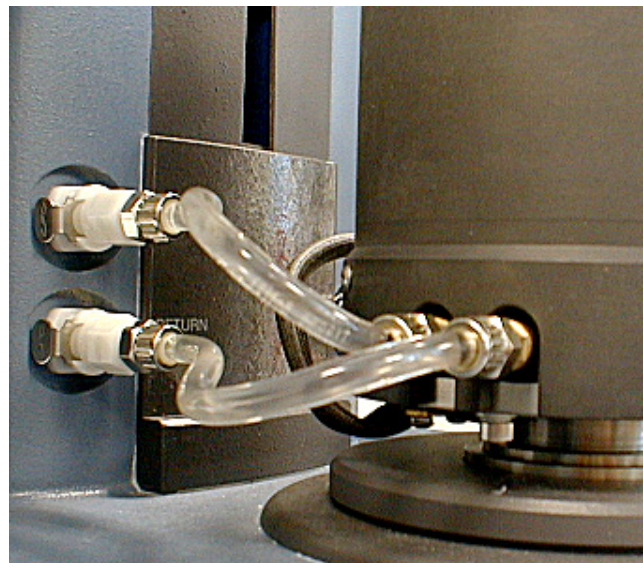


Figure 5.12
Connecting Fluid Cables

7. Lower the rheometer head until the datum mark on the shaft of the rotor is level with the top of the cup as shown in Figure 5.13 to the right. You can now set up the measuring geometry in the rheometer software and set the gap explained in the online help.

Changing the Cup

If you need to change the size of the cup you are using, follow these steps:

1. Undo the two screws on the cup. Turn and lift it out as shown in the figure below.
2. Replace with the required cup size and twist into place. Tighten the two screws by hand.

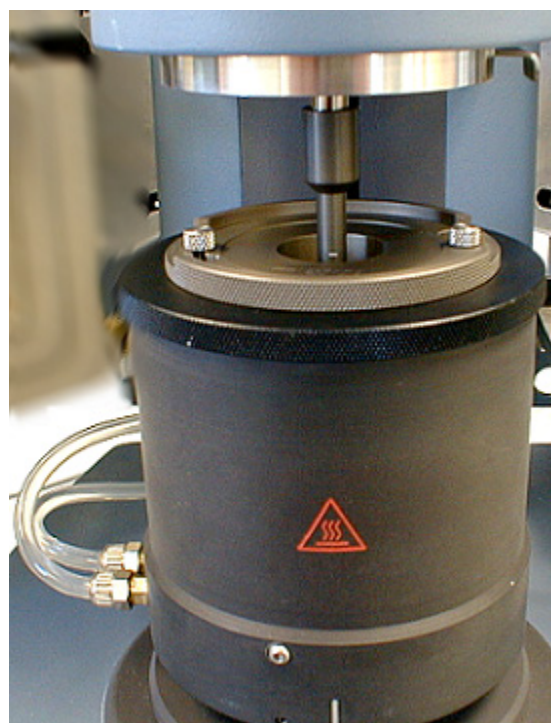


Figure 5.13
Lowering Rheometer Head

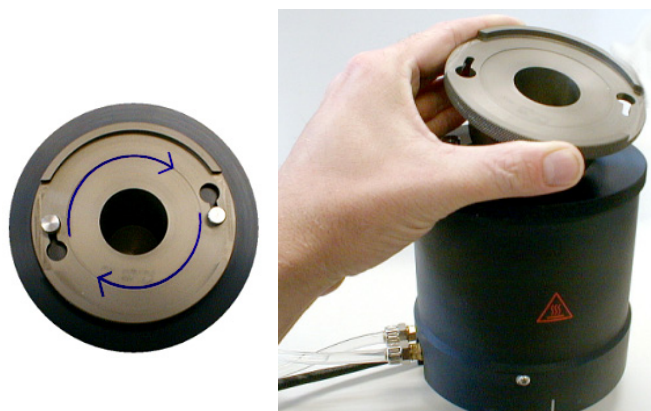


Figure 5.14
Changing the Cup

Using the ETC

This section provides information on how to install and set up the Environmental Testing Chamber (ETC). For more information on the ETC, see the Rheology Advantage online help.

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 (see the start of this chapter).
3. Turn off the power to the rheometer control box.
4. Ensure that the two top screws (A and B in Figure 5.15) are fitted with washers and are located in place—but make sure that they are almost totally unscrewed (two turns in).
5. Open the ETC oven (see Figure 5.16) and then use the handles on the oven doors to lift it onto the two top screws. Lightly tighten these screws.

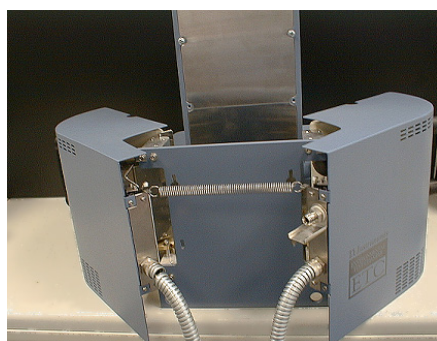


Figure 5.16
The ETC Open

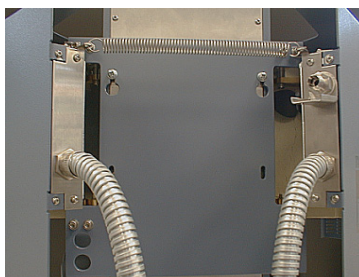


Figure 5.15
Mounting Screws

6. Insert the final two screws (C and D in Figure 5.15).
7. Adjust the position of the ETC on the screws and then tighten all four.
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 Figure 5.17.
10. Open the ETC oven doors to gain access to the Smart Swap™ mounting.

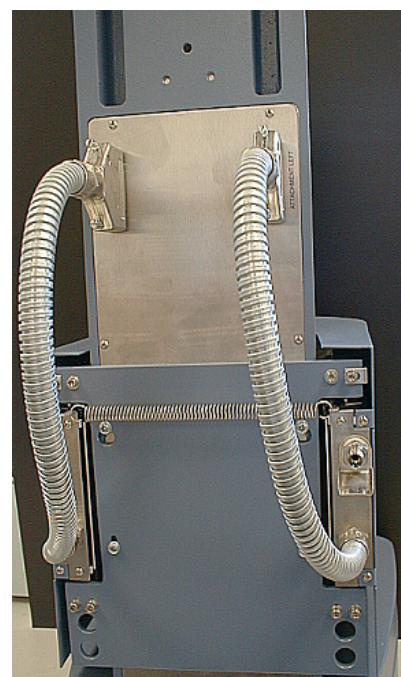


Figure 5.17
Connecting the Cables

11. 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.

Le déverrouillage restera actif pendant 10 secondes.

12. Fit the lower attachment, ensuring it is aligned correctly. See Figure 5.19 below.



Figure 5.19
Fitting the Lower Attachment

13. Connect the cable from the lower attachment to the rheometer as seen in Figure 5.20.

14. Close the oven and ensure that no part of the doors touch any part of the lower fixture. Adjust the position of the ETC again, if required.

15. Attach the upper geometry, again making sure that no parts are touching the fixture, adjusting the ETC if necessary.
16. If you plan to use the liquid nitrogen option with the ETC, skip the following steps and proceed to the next section for installation instructions.
17. Insert the shorting plug into the Event A connection on the rheometer as shown here.

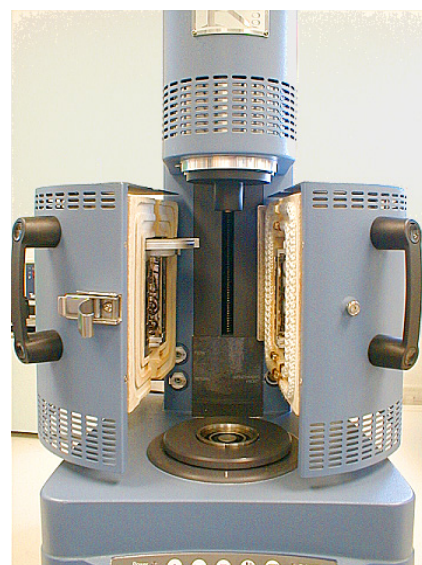


Figure 5.18
ETC with Open Oven Doors



Figure 5.20
Connecting the Cable from the Lower Attachment to the Rheometer

18. Connect the purge gas to the rheometer as seen in Figure 5.21 to the right. If you have a suitable supply of nitrogen gas (2 bar minimum pressure, nominal 10 liters per minute flow rate) it is recommended that you connect the feed gases to the ETC as shown in Figure 5.23. Otherwise, connect as shown in Figure 5.22 below.



CAUTION: The reducing valve is factory-set to 10 liters per minute and should not be adjusted.

La valve réductrice est réglée à 10 litres par minute et ne devrait pas être modifiée.

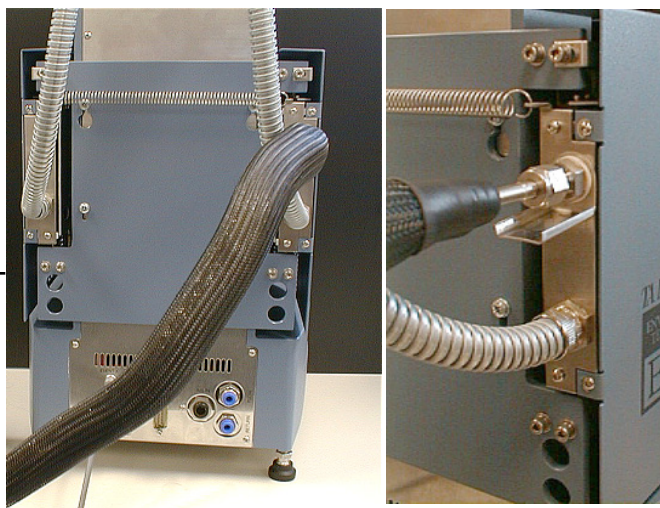


Figure 5.21
Connecting the Purge Gas

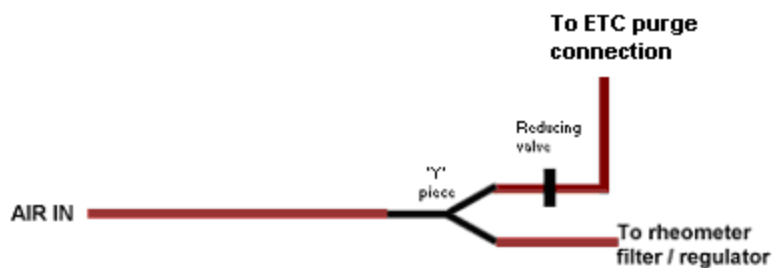


Figure 5.22
ETC Connections Using Air as the Agitation Gas

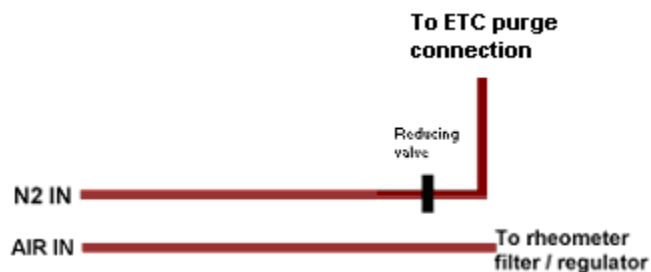


Figure 5.23
ETC Connections Using Nitrogen as the Agitation Gas

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

Installing the Low Temperature Accessory

In order to operate the ETC at temperatures below ambient and also to facilitate rapid cooling, the (optional) low temperature accessory can be used. This works by supplying a controlled flow of liquid nitrogen/cold nitrogen that is fed down the inside of the oven and evaporates off the wire wool.

Follow the installation procedure in the previous section up to step 15. Then use the following additional steps to complete the installation (see Figure 5.25 and Figure 5.26).

1. Connect the Event cable from the flow control assembly to the Event A connection on the rheometer.
2. Ensure that the cryogenic system has been installed as directed in the instructions supplied by the manufacturer.
3. Connect the flexible hose from the outlet of the cryogenic cooling system to the '**Liquid in**' connection on the flow meter assembly as shown in Figure 5.24. Connect the purge gas from the flow control assembly to the rheometer.

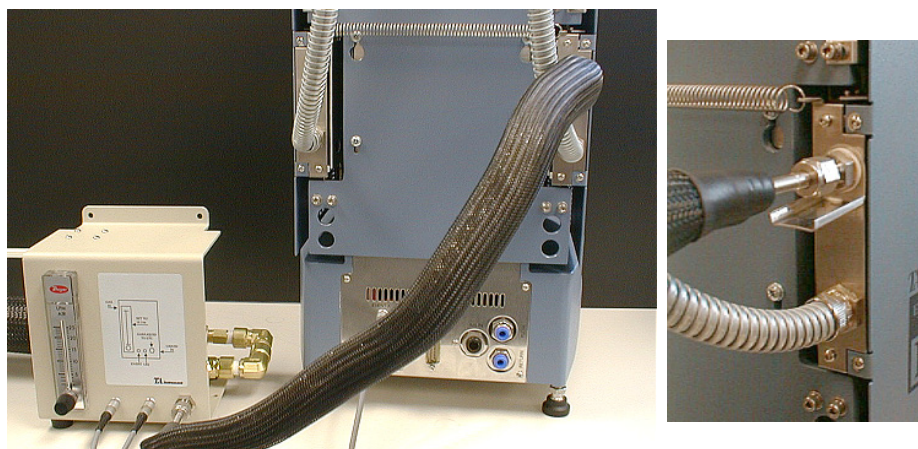
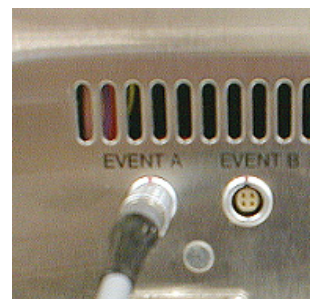


Figure 5.24
Connecting the Hose

4. Connect a gas feed to the '**Gas in**' connector on the flow control assembly. If you have a supply of nitrogen gas follow Figure 5.26, otherwise follow Figure 5.25.
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 liters per minute (LPM) on the flow meter assembly.

* 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 "Operating Hints" later in this chapter.

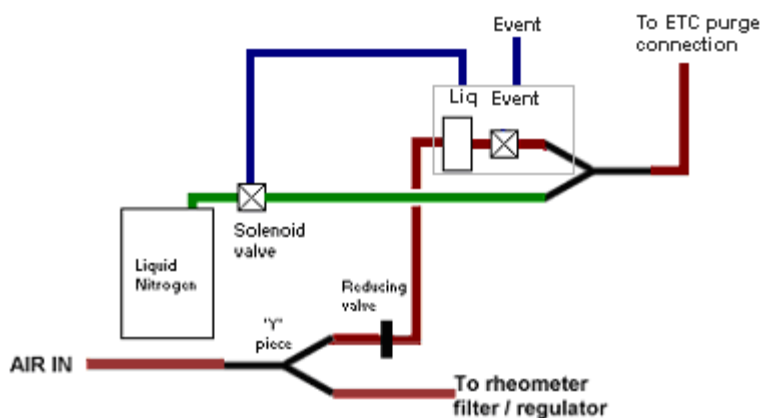


Figure 5.25
ETC Connections with LN2

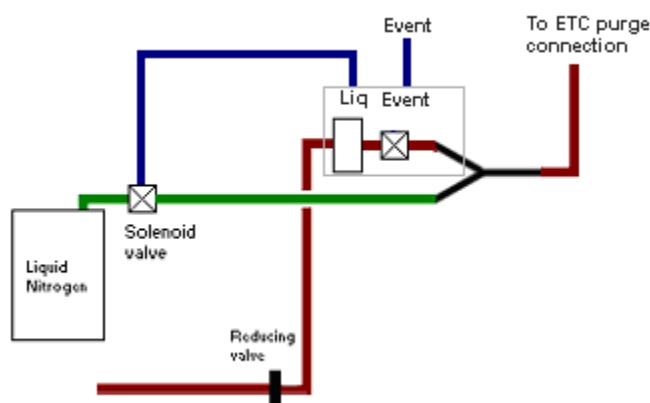


Figure 5.26
ETC Connections with Nitrogen

If the system has been set up according to the instructions, the rheometer should now be ready to use. However, we recommend that you follow a few extra precautions, described on the next several pages.

- 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. The Rheology Advantage™ 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, or replace them with a fresh pair. 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.
- 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. (Further information on the mapping procedure can be found in the Rheology Advantage Help™ system.)

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.



WARNING: The electronic control box supplied with the ETC has no user serviceable parts inside.

Le cadre de commande électronique fourni avec le four ne contient aucun consommable.

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."

General Operating Guidelines

To ensure that your temperature system operates efficiently and safely, follow these suggestions below.

Do

Ensure that all operators of this equipment have been correctly trained and are aware of the safety information contained in this manual.

- Put this list (or a similar one) in a prominent place near the instrument.
- Read all instruction manuals supplied, as they contain useful operational hints and maintenance information.
- Ensure that the gap is correctly set.
- When installing parallel plate geometries, carefully ensure that the thread is engaged squarely to avoid the possibility of cross threading.
- Avoid any unnecessary movement of the liquid nitrogen carrying hoses when at low temperatures. Excessive movement or strain could cause the hose to crack.

Do Not

- Leave the high temperature system switched on or the nitrogen tank tap open, when not in use.
- Attempt to remove a hot geometry without wearing safety gloves.
- Forcibly remove a geometry.
- Allow any object to obstruct the safety interlock sensors at the rear of the ETC housing.

Keypad Functionality

The following table provides a list of each button found on the instrument keypad shown in the figure below.

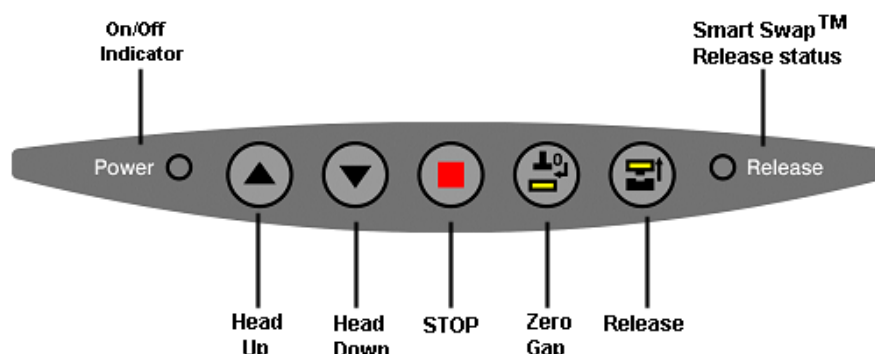


Figure 5.27
AR 2000 Keypad

Feature	Description
On/Off Indicator	A continuous red light indicates that rheometer is receiving power.
Smart Swap Release Status	
<i>Off</i>	Attachment holder is locked.
<i>Flashing green</i>	Power to attachment is removed (holder still locked.)
<i>Continuous green</i>	Attachment holder is unlocked.
Head up	Moves the rheometer head up while pressed.
Head down	Moves the rheometer head down while pressed.
STOP	Aborts the current activity on the rheometer, such as gap zeroing, running a procedure etc.
Zero Gap	Initiates an "auto-zero" of the gap using the currently installed measuring geometry. This duplicates the functionality of the zero gap button in the instrument software.
	To maximize gap zeroing time, you should position the geometry to within 5 mm above the plate before pressing this button.
Release	Activates the release mechanism for Smart Swap (see previous page for more information).

Levelling the Rheometer

Optimum performance depends upon the instrument being level and in a sturdy position to avoid the possibility of rocking. To check and see whether your instrument is level, simply place a bubble spirit level *on the lower temperature module plate*.

If the instrument is not level, screw the adjustable feet (located at each corner of the instrument) either in or out, as necessary. Check the spirit level after each adjustment. Once you have the instrument levelled correctly, press each corner of the instrument to check that all four feet are in contact with the laboratory bench. Any movement caused by pressing should be rectified by adjusting the feet, and then rechecking the level of the plate.

If the spirit level is the circular type, it should be placed in the middle of the plate. If the spirit level is the bar type, place it along a diameter of the plate. Check the level by placing it along another diameter of the plate at 90° to the first position.

Checking Your System

After installation has been completed, start the instrument to check to make sure everything is working and that all parts of the system are communicating with each other. Use the following steps to check your system:

1. Turn on the air supply to the instrument.
2. Turn on the water supply to the instrument.
3. Remove the air-bearing clamp.
4. Turn on all electrical parts of the system (rheometer, PC, etc.). A system check will be initiated as shown by the LCD on the electronics control box.
5. Start the rheology software.
6. Select the Instrument Status screen in the software.
7. If everything is installed correctly, the instrument will display continually updating figures.
8. Lower the head using the buttons in the software. If the installation is OK, the head will operate.
9. Input a temperature slightly different to that displayed. If the installation is OK, the temperature will change to the new one you have just input.
10. Raise the head.

If all of these actions result in the correct response, you can be confident that you have installed the system correctly and it is ready for use. If you have problems, please contact your local TA Instruments office or their appointed agent.

Calibrating the Rheometer

Strictly speaking, you cannot calibrate your rheometer yourself. You can check that the instrument is functioning properly by measuring the viscosity of a certified standard Newtonian oil (PTB 1000A nominal 1 Pas at 20.0°C). If you get a greater than 4% error in the reading, there is a possibility that your rheometer needs some attention from a TA Instruments Service Engineer.

Carry out the following experiment:

1. Attach a 60 mm 2° stainless steel cone to the rheometer. (This is the preferred geometry, if you do not have one use the largest cone that you do have.)
2. Set the zero gap and measurement system gap in the usual way.
3. Carefully load the sample ensuring correct filling.
4. Carry out a 4-minute flow test, continuous ramp, controlled stress range 0 to 88.0 Pa at 20°C.
5. Determine the Newtonian viscosity. If this value is more than 4% different from the certified value, repeat the experiment. If there is still an error, call your local TA Instruments office for advice.

There are several sources of operator error that can give erroneous answers. This does not necessarily mean that your instrument is not working properly. These include errors in setting the gap, incorrect temperatures used, or over- or under-filling of the gap. This calibration check needs to be carried out monthly.

Shut-Down Procedure

When you are ready to turn the instrument off, it is important that you follow the steps listed below in the correct order.

1. Raise the head and remove the measuring geometry.
2. Exit the software package that you are currently running.
3. Turn off the rheometer and the computer.
4. Replace the air-bearing clamp.
5. Turn off the water supply to the instrument.
6. Turn off the air supply to the instrument.

Cleaning the Filter Regulator Assembly

The air bearing requires a very clean supply of air regulated to a stable pressure of between 25 to 40 psi, dependent upon the air bearing. The filter regulator assembly is an important part of your rheometer system. It is designed to meet the required standards of cleanliness (99.9999% of particles above 0.01 mm retained) and regulation, given that the source of air is dry and pre-filtered.

The maximum inlet pressure is 147 psi (10 bar). The maximum pressure to the rheometer is 42 psi (3 bar).

The filter regulator assembly is shown schematically in Figure 5.28.

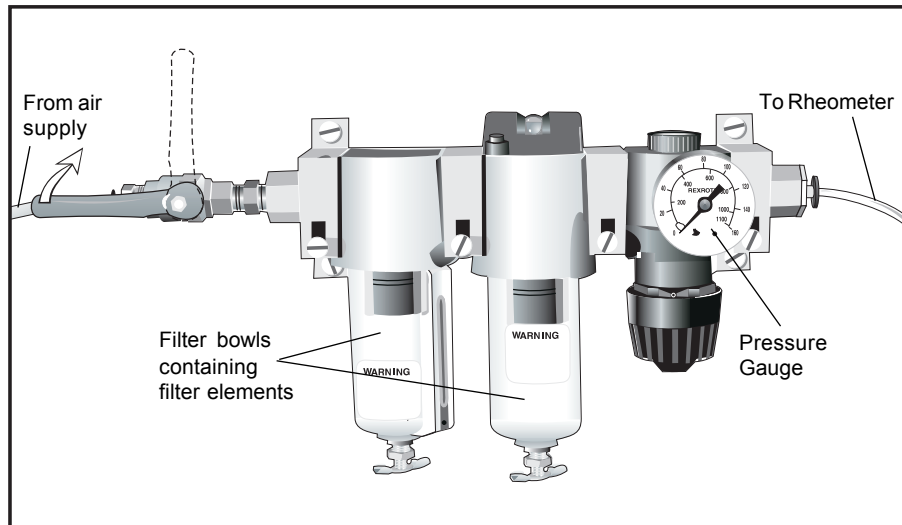


Figure 5.28
The Filter Regulator Assembly

If you use the filter regulator, you will need to check routinely (*i.e.*, at least monthly) for any signs of contamination (*i.e.*, water, oil or dirt) collecting in the filter bowls. If you see a build up of water, follow these steps:

1. Turn off the air supply and disconnect the assembly from the rheometer. Remember to put the airline plug into the back of the rheometer.
2. Unscrew the filter bowl plug and dry the inside thoroughly.
3. Replace the plug and purge with air before reconnecting to the rheometer. The filter elements must also be replaced when there is a visible buildup of dirt.

Chapter 6

Measuring Systems

Overview

This chapter describes the geometries currently available from TA Instruments and provides guidelines to choose the optimum geometry (or measuring system) for each application. A complete geometry catalog is available which describes in detail each geometry and typical applications. Contact your local TA Instruments office or their appointed agent for further details. Some theoretical considerations are given that will provide guidance and help you maximize the use of the AR Rheometers.

TA Instruments offers a range of geometries. The geometries are divided into the following groups, each with a range of sizes available:

- Cone and plate
- Parallel plate
- Concentric cylinders.

The following pages describe the types of geometries and provide details on how to attach the geometry to the rheometer.

General Description

The measuring system is defined as those parts that are in direct contact with the sample or material.

A measuring system consists of two parts:

- One is the fixed member (or Stator), for example, the Peltier plate.
- The second part (the geometry) is attached to the driving motor spindle, where it is locked in position using the draw rod. The draw rod is detachable and passes through the centre hole bored in the spindle. The geometry constitutes the moving member of the system (the Rotor).

Geometry Materials

Geometries are usually constructed from stainless steel, aluminium, or acrylic (other materials can be supplied upon request). The rotor should ideally be as light as possible to minimize inherent inertia effects. It should also be chemically compatible with the test sample in order to avoid corrosion problems.

Stainless Steel

Stainless steel is relatively heavy, but it has a low coefficient of thermal expansion. It is compatible with most test materials and is robust enough to withstand heavy use, even if you are a less experienced operator.

Aluminium

Aluminium has a higher thermal coefficient of expansion and is limited because of its chemical compatibility. As it is lighter, inertial effects are not as great.

Plastic

Engineering grade acrylic, polycarbonate, and rigid PVC are all suitable materials for geometry construction. These are transparent so the visual behavior of the sample can be observed. Plastic geometries are also much lighter than metallic geometries.

Acrylic and polycarbonate have less inertial problems as they are relatively light, but they have limited chemical compatibility. You should not use plastic geometries above 40°C.

Cone and Plate

A schematic of a cone and plate system is shown below in Figure 6.1. It is important to know how to calculate the stress and shear rate factors for each geometry before deciding on the geometry dimensions.

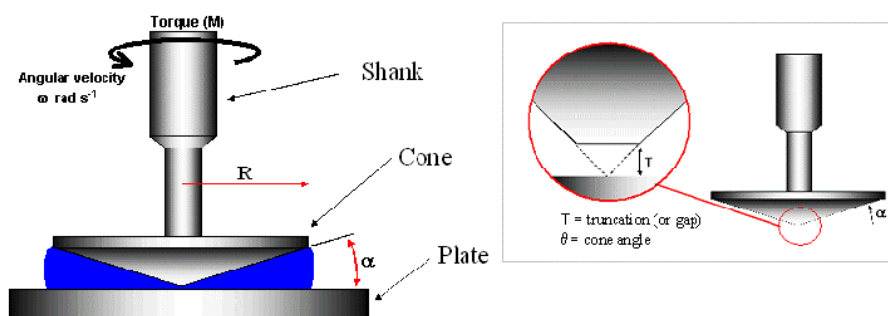


Figure 6.1
The Cone and Plate

$$\text{Shear rate (s}^{-1}\text{)} = F_{\dot{\gamma}} \omega$$

$$\text{where } F_{\dot{\gamma}} = \frac{1}{\tan \alpha}$$

$$\text{Shear stress (Pa)} = F_{\sigma} M$$

$$\text{where } F_{\sigma} = \frac{3}{2\pi R^3}$$

The standard diameters available are 20 mm, 40 mm and 60 mm with cone angles of 0.5° to 4° in 0.5° increments.

Cone and plate geometries are generally used for single-phase homogeneous samples or samples with submicron particles. Samples containing particulate matter are usually unsuitable for cone and plate geometries as the particles will tend to migrate to the apex of the cone and will get jammed in the truncation area. Erroneous data will result.

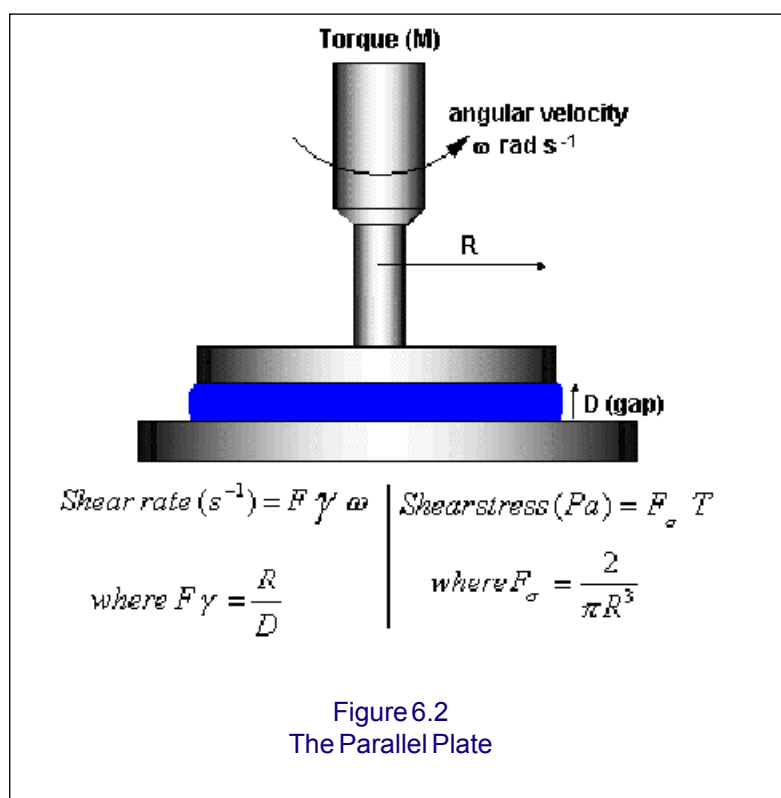
The angles and truncation values of each cone are individually calibrated. A calibration certificate is available. The serial number, angle and truncation are all inscribed on the stem of each cone.

Parallel Plate

The parallel plate system allows samples containing particles to be effectively measured. You can set the gap to any distance, thereby eliminating the problems due to particles size. A good rule of thumb for particulate materials is to set a gap size set at least 10 times greater than the largest particle size. For example, if the maximum particle size is 100 μm , you should set the gap to at least 1000 μm .

The main disadvantage of a parallel plate system is that the stress is not uniform across the entire diameter. However, the software compensates for this fact. The shear stress and shear rate factors given are with respect to the rim.

A schematic of a parallel plate is shown in Figure 6.2.



Concentric Cylinders

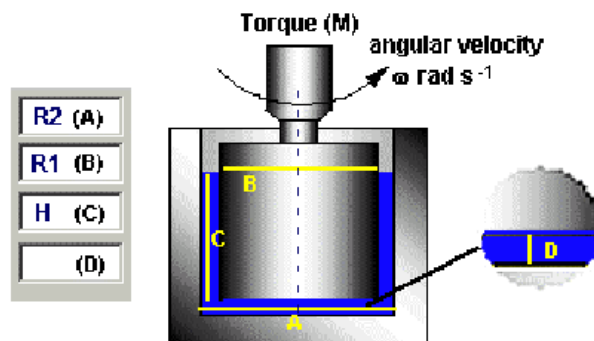
Concentric cylinder systems (or cup and bob) are generally used for lower viscosity samples that may not be held within the gap of cone and plate or parallel plate systems. (See later in this chapter for information on setting up and using the concentric cylinder systems.)

There are several different types including:

- Recessed end
- Conical end (DIN)
- Vane
- Double concentric.

See the following figures for examples of concentric cylinder geometries.

The previous equations are also used for the Vane system.

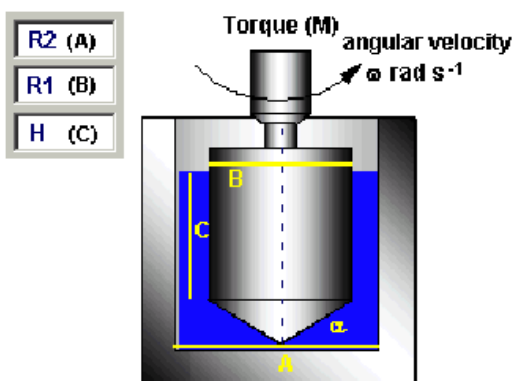


Assuming no end effects.

$$\text{Shear rate (s}^{-1}\text{)} = F \gamma \omega \quad \text{Shear stress (Pa)} = F_{\sigma} M$$

$$\text{where } F \gamma = \frac{R_2^2 + R_1^2}{R_2^2 - R_1^2} \quad \text{where } F_{\sigma} = \frac{R_1^2 + R_2^2}{4\pi H R_1^2 R_2^2}$$

Figure 6.3
Recessed End

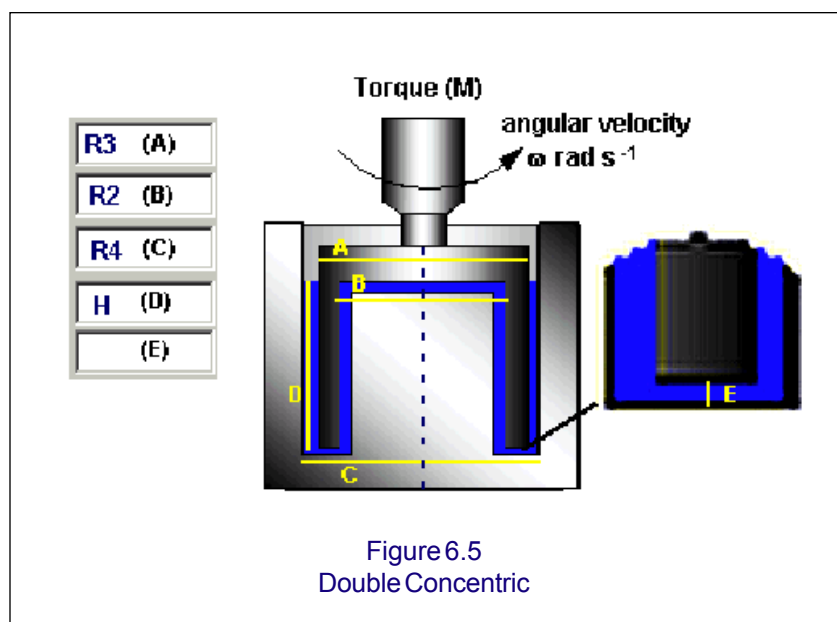


$$\text{Shear rate (s}^{-1}\text{)} = F \gamma \omega$$

$$\text{where } F \gamma = \frac{R_2^2 + R_1^2}{R_2^2 - R_1^2} = \tan^{-1} \alpha$$

Figure 6.4
Conical End

The shear stress factor is the same as the geometry shown in Figure 6.3. The conical end aids penetration and even distribution of stiffer samples.



The ratio $R_1:R_2 = R_3:R_4$. The shear rate is then calculated as in Figure 6.3 using R_3 and R_4 .

$$\text{Shear stress (Pa)} = F_{\sigma} M$$

$$\text{where } F_{\sigma} = \frac{R_1^2 + R_2^2}{4\pi H R_2^2 (R_1^2 + R_3^2)}$$

Using the Stress and Shear Rate Factors

The TA Instruments operating system software calculates the stress and shear rate factors, which are used by the software in all subsequent calculations.

However, there may be occasions when you will need to enter these factors manually. If you do, follow the sequence given below:

1. Multiply the angular velocity (ω) by the shear rate factor ($F_{\dot{\gamma}}$) to obtain the shear rate (s^{-1}).
2. Multiply the angular displacement by the same factor to obtain the strain (dimensionless).
3. Multiply the torque (T) (μNm) by the shear stress factor (F_{σ}) to obtain the shear stress (Pa).

Choosing the Best Geometry

When selecting the correct measuring geometry to use, it is important that you understand the following:

Exactly what type of experiment do you wish to carry out?

What is the sample behavior like—does the sample contain particles?

And probably most importantly, what is the real-life situation are you trying to recreate?

Sometimes the answers to all of the above questions are not known, but there are some basic guidelines that will help you. However, it is also important to remember that you are measuring the bulk properties of the material itself, and this should be independent of the type of geometry used (within reason!).

Cone and Plate/Parallel Plate Systems

The cone and plate and parallel plate systems both need small sample volumes, are easy to clean, have low inertia, and can potentially achieve high shear rates. The additional advantage to using a cone and plate is that the shear rate is uniform throughout the sample, and the parallel plate can accommodate large particles.

Generally, the cone and plate or parallel plate systems can be used for almost any sample. They are easy to set up and use, making one of these systems the best choice for optimum results. They are both available in different sizes, therefore, it is important to understand how to choose the system with the correct dimensions.

Angles

Cones are supplied by TA Instruments in any angle from 0° to 4° , usually in 0.5° increments. The 4° cone is the largest available, as the sample velocity profile becomes unpredictable at higher angles and the mathematical expression of $\alpha \simeq \tan \alpha$ is no longer valid. The 4° cone is ideal for creep measurements, because a longer displacement is required per unit strain.

The smaller the angle (or gap in a parallel plate system), the higher the maximum shear rate obtainable.

Diameters

The *smaller* the diameter of a cone or parallel plate system, the *larger* the shear stress factor. This means that a small (e.g., 20 mm) diameter geometry should be used with stiffer materials or medium to high viscosities. A 40 mm geometry is more versatile and it usually allows the majority of medium viscosity materials to be measured.

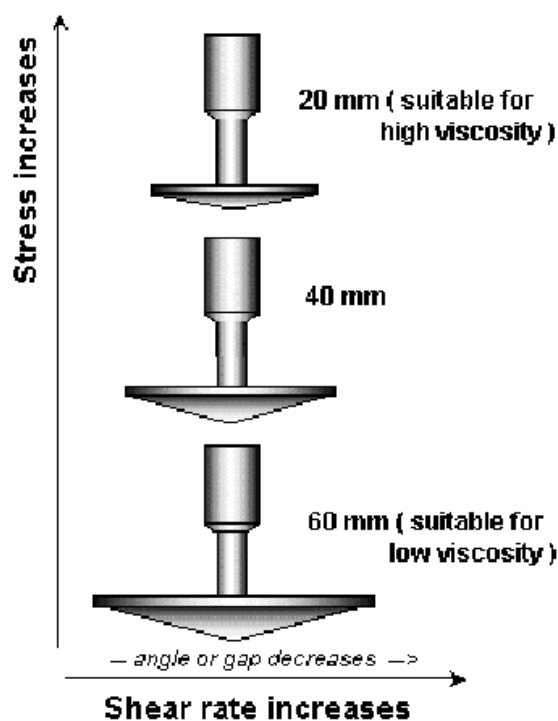


Figure 6.6
Choosing Geometry Angle and Diameter

A large diameter geometry (*e.g.*, 60 mm) is more sensitive to stress changes and is used to measure low viscosity samples.

Be sure to load the sample correctly and be careful not to under or overfill the geometry. If this occurs, it would effectively change the diameters of the cones and, hence, adversely affect the shear stress factors.

Figure 6.6 summarizes the information given above on the choice of angle (or gap) and diameter.

Material

Stainless steel is relatively heavy, has a low coefficient of thermal expansion, is compatible with most samples, and is very robust.

Aluminium geometries are lighter than steel, but have a larger coefficient of thermal expansion. They will go to temperatures greater than 40°C, but are still heavier than acrylic.

Acrylic geometries are very light and are, therefore, most suitable to use with low viscosity samples. However, you should not use acrylic geometries above 40°C.

See the beginning of this chapter for more details on materials.

Preventing Solvent Evaporation

If you are using samples that contain volatile solvents or are water-based, evaporation can cause problems during measurements. TA Instruments has overcome this problem by using a solvent trap cover, which sits over the geometry (but does not touch it).

Solvent trap version geometries have a well on top of the geometry. Place a small amount of the relevant solvent into this well. The solvent trap cover has a lip that sits in the solvent, allowing the free space around the sample to become saturated with the solvent vapor, which prevents evaporation.

A schematic of a solvent trap cover and geometry is shown in Figure 6.7 below.

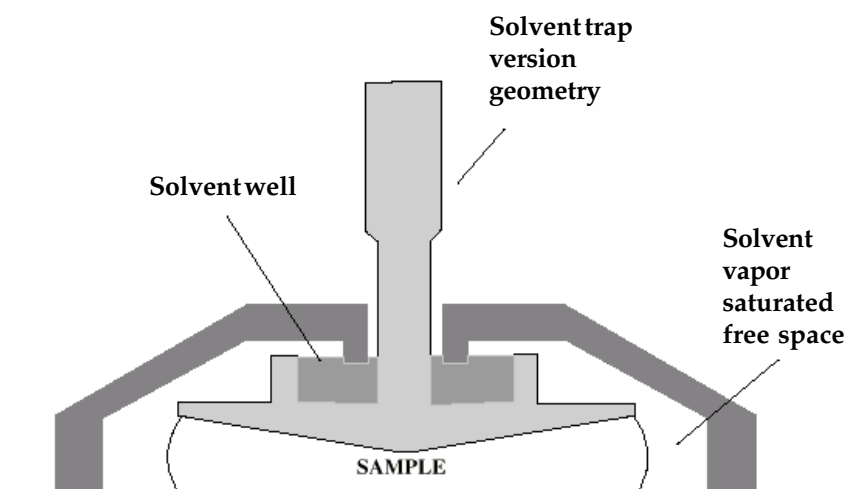


Figure 6.7
Solvent Trap Cover and Geometry

When using solvent trap systems it is generally advisable to run the inertia correction wizard (with solvent in the trap, but no sample loaded).

Preventing Slippage at Sample/Geometry Interface

Some samples, such as hydrogels, contain a lot of water that can migrate to the surface of the sample. This can cause a film layer to form between the bulk of the material and the geometry surface, causing slippage at this interface. To alleviate this problem, use special crosshatched geometries, which, in effect, have the measuring surface slightly roughened. (However, when you use these *crosshatched* geometries, there is a trade-off between absolute accuracy and repeatability.)

Removing the Air-Bearing Clamp

You should *never* remove the air-bearing clamp until the air supply is connected and switched on.

Once the air supply is switched on, and you can hear the air through the rheometer, the clamp can be safely removed.

To remove the clamp, firmly hold the clamp and unscrew the draw rod by turning it counterclockwise (anticlockwise).



CAUTION: Always hold the clamp and turn the knob - never the other way round.

Toujours tenir la géométrie et tourner la molette - jamais le contraire.



Figure 6.8
Removing the Air Bearing Clamp

Next, slide the bearing lock away and ensure that the bearing is free to rotate.

The clamp is replaced in exactly the same way. The air must not be switched off until the clamp is in place.

If your instrument does not have a bearing lock, then ignore this step. This mechanical lock has been removed from current production unit as the software Bearing Lock implemented with the Mobius Drive provides the same function without the risk of leaving it in place at the start of a measurement.

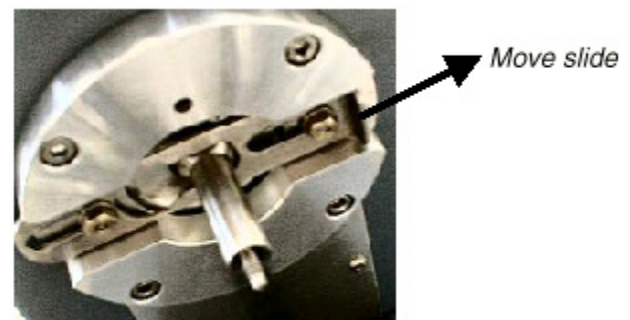


Figure 6.9
Slide Lock Away

Attaching a Geometry

This procedure is carried out using the same technique as described for the air-bearing clamp:

1. Switch on the air and remove the air-bearing clamp by turning the draw rod counterclockwise (anticlockwise).
2. Push the geometry up the drive shaft and hold it while placing the draw rod in the screw thread of the geometry.
3. Screw the draw rod upwards (clockwise). It should be screwed finger tight, but not forced.

To remove the geometry, use the reverse process.

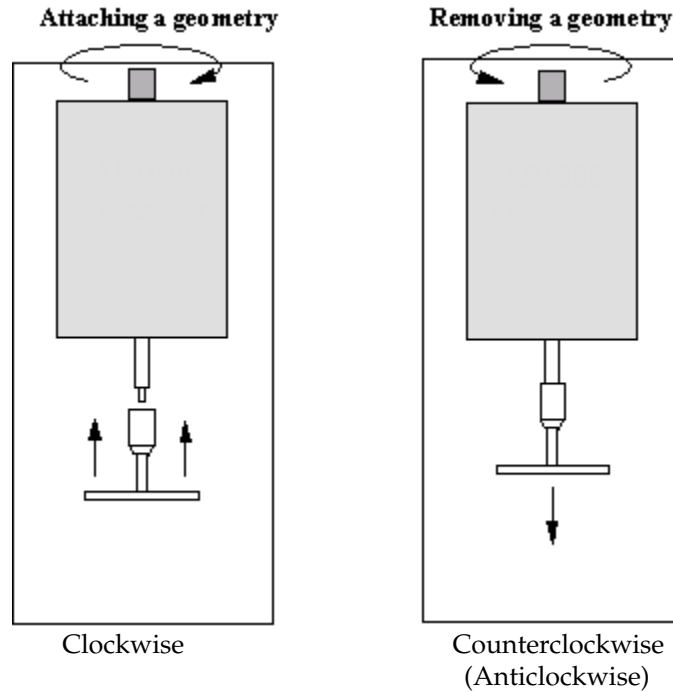


Figure 6.10
Attaching/Removing A Geometry

Ensuring that the Sample is Loaded Correctly

Ensuring that the sample is loaded correctly and the gap is properly filled is probably one of the most important points to consider in any rheological experiment.

You will find that you will become quite adept at judging the right amount of sample to use, depending upon the geometry diameter and gap size. You can either calculate the exact volume or weight of sample needed. However, care must be taken if you intend to use a pipette or syringe to deliver the correct amount. Samples that are delicately structured will be adversely affected by the high shear rate regime encountered in syringes or pipettes. If the gap is not filled correctly, there are certain types of errors that can occur. The magnitude of the errors will be entirely sample dependent, but generally over filling is less of a problem than under filling. Such errors are called *edge effects*. Figure 6.11 shows the different types of filling encountered.

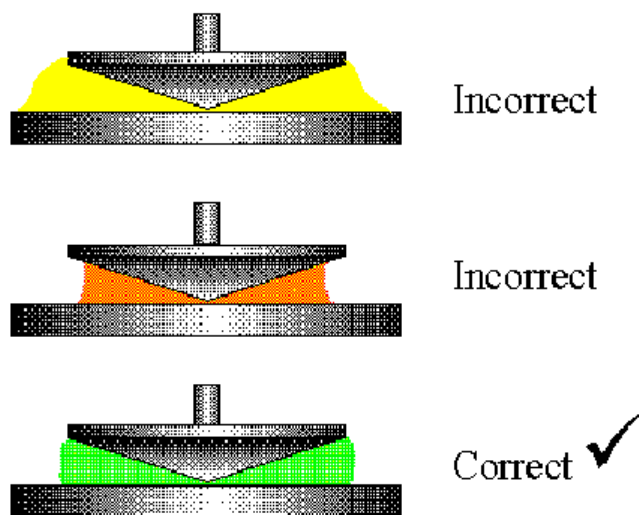


Figure 6.11
Loading the Sample

- If the gap is *overfilled*, some of the excess sample may migrate to sit on top of the geometry. If, however, the sample is of low viscosity, this is not likely to happen and the errors are reduced.
- If the gap is *underfilled*, you are effectively altering the diameter of the geometry. This will inevitably introduce large errors and you should definitely avoid this situation.

Loading the sample correctly is a skill that is learned with time. It may help you to spend some time initially simply loading and reloading a sample. The correct loading is vital to accurate and meaningful results.

Chapter 7

Using the Upper Heated Plate

Introduction to the Upper Heated Plate

At the standard AR 2000 Smart Swap™ Peltier plate's temperature range extremes, a 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) has been developed to allow this and is used in conjunction with the standard Smart Swap™ Peltier plate.

The Upper Heated Plate 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 Figure 7.1). Heating of the Upper Heated Plate is through the electrical elements.

Cooling is provided by water or other fluid carried in the coolant channel.

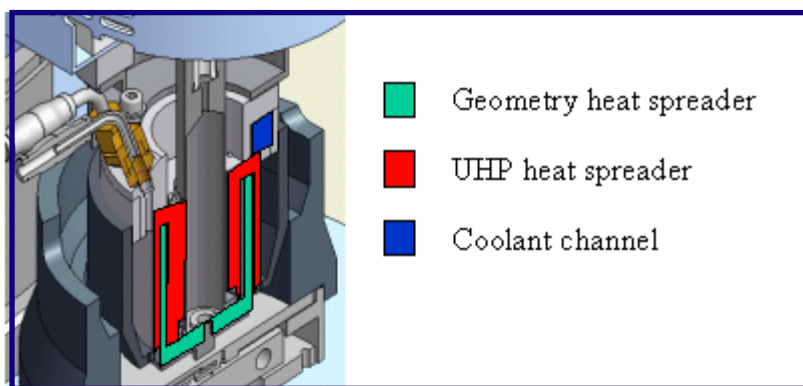


Figure 7.1
Exploded View of the UHP and Upper Geometry Holder

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 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.

Attaching the Upper Heated Plate to the AR 2000

Follow the steps below to attach the Upper Heated Plate to the AR 2000 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 (see Figure 7.2).

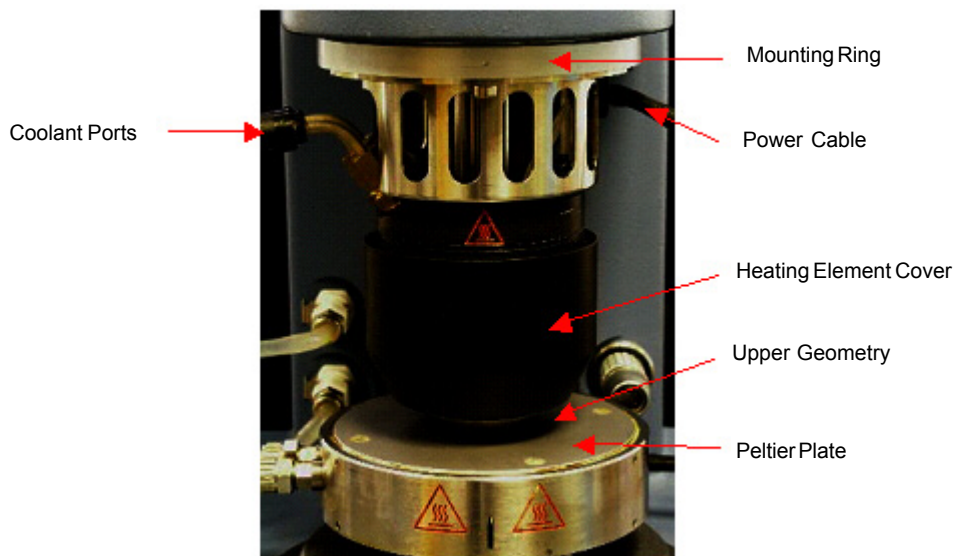


Figure 7.2

The Upper Heated Plate Shown Mounted on an AR 2000 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 adaptor respectively (see Figure 7.3 to the right).

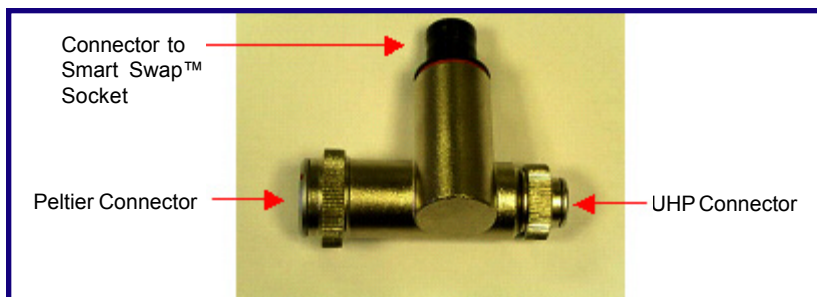


Figure 7.3

The Smart Swap™ UHP Adaptor

5. Connect the Upper Heated Plate adaptor to the Smart Swap™ socket (see Figure 7.4 to the right).
6. To return the temperature control to Peltier plate only, remove the adaptor from the Smart Swap™ socket using the “Release” button on the instrument control panel. Remove the Peltier connector from the adaptor and plug the connector directly into the Smart Swap™ socket.



WARNING: Do not remove the heating element cover.

ATTENTION: N'enlevez pas la couverture d'élément de chauffe.



Figure 7.4
Connection of the UHP Adaptor to the Smart Swap™ Socket

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, www.julabo.com; mains water supply is also normally suitable). Note that if a single supply is used, the Peltier and Upper Heated Plate should always be connected in **parallel**, never in series. 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.

Important: Pour une opération efficace, le plan de Peltier et l'Upper Heated Plate devraient être reliés en parallèle, PAS en série, si la même source en eau est employé pour tous les deux.

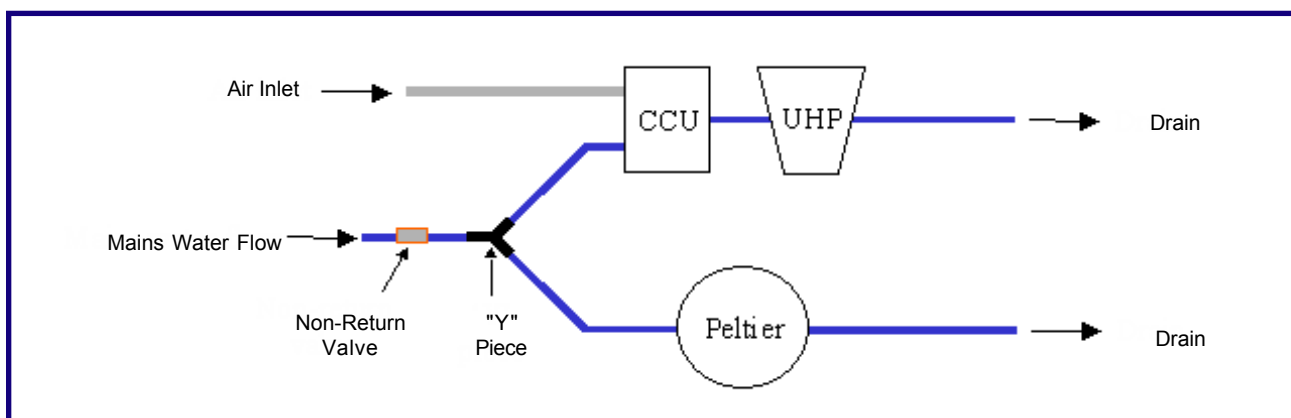


Figure 7.5
Cooling Water Configuration 1
Mains Water Supplying Both Peltier and Upper Heated Plate

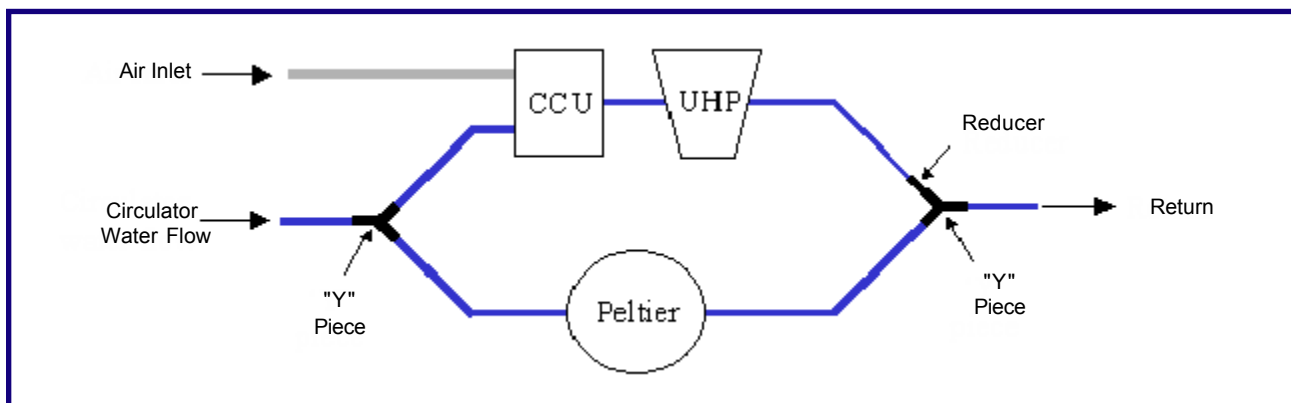


Figure 7.6
Cooling Water Configuration 2
Fluid Circulator Supplying Both Peltier and Upper Heated Plate

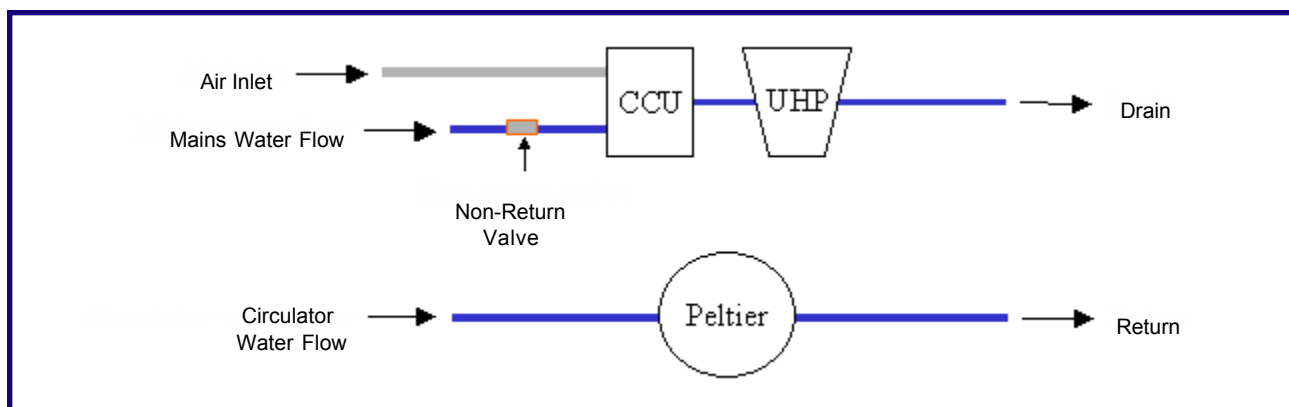


Figure 7.7
Cooling Water Configuration 3
Fluid Circulator Supplying Peltier, Mains Water Supplying Upper Heated Plate

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 may be free standing, or wall mounted using the clearance holes on top of the unit (see Figure 7.8 to the right).

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 (see Figure 7.9 below) should be placed in the line upstream of the CCU.

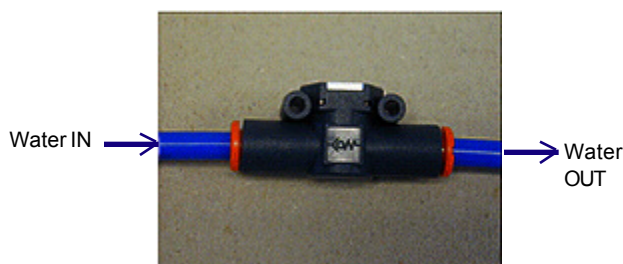


Figure 7.9
Non-Return Valve
(For use with mains water supply only.
Note the direction of flow through the valve.)



Figure 7.8
The Cooling Control Unit

Important: Note the direction of flow through this valve.

Important: Notez la direction d'écoulement à travers de la 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 adaptor provided.

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 adaptor and 6-mm "Y" piece are provided for the connection to the fluid circulator.

Coolant Inlet Port

Inert Gas Port

Coolant Outlet Port

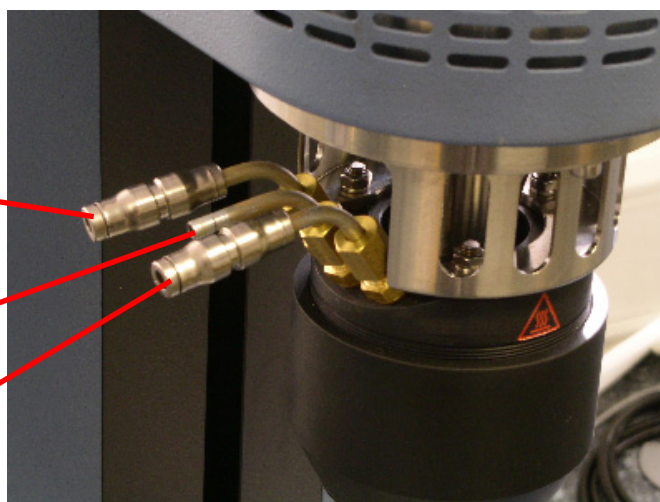


Figure 7.10
Coolant and Inert Gas Connections for the UHP

5. Connect the EVENT socket on the CCU to the EVENT B socket on the rear of the AR 2000 Rheometer, using the cable provided.
6. Set the purge air flow rate to 1 Liter per minute (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 Rheology Advantage™ software, to ensure continuous air flow.

Using Circulating Fluids Other Than Water

For low temperatures, circulating fluids other than water must be used. 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.

ATTENTION: Des fluides inflammables tels que l'éthanol ou les huiles minérales ne devraient pas être employés avec l'Upper Heated Plate. Des fluides de circulation ne devraient pas être employés en dehors des gammes données 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 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 valve bracket to the EVENT B socket on the rear of the AR 2000 rheometer, using the cable provided.

Table 7.1 shows minimum and maximum temperatures for the Upper Heated Plate, using circulating fluids available from Julabo GmbH, www.julabo.com, with an FP50-MS fluid circulator supplied by the same company.

Table 7.1

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.

ATTENTION: Le montage d'Upper Heated Plate, le support supérieur de la géométrie et la géométrie supérieure, peuvent être chauds. Assurez-vous que ces composants sont froids avant d'essayer d'enlever ou remplacer le support de la géométrie supérieure.

Connecting the Geometry and Holder

To connect the upper geometry and holder follow these steps:

1. Raise the instrument head fully, using either the Rheology Advantage™ software or the **Head UP** button located on the instrument control panel.
2. Attach the geometry to the holder, using the attaching tool provided, if necessary. (This tool cannot be used with the 40-mm diameter geometry, which can be attached to the holder by hand.)
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.

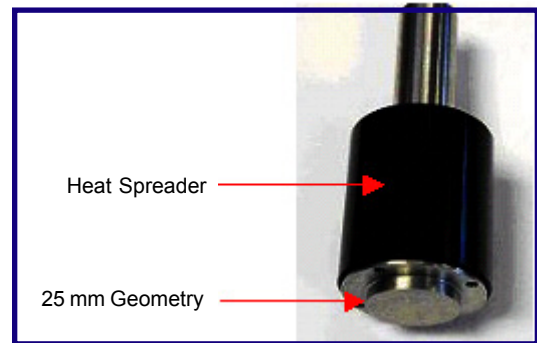


Figure 7.11
Upper Geometry Holder
(Shows the cylindrical heat spreader with a 25 mm diameter geometry in place.)

Removing the Geometry and Holder

To remove the upper geometry and holder from the AR 2000 rheometer follow these steps:

1. Raise the instrument head fully, using either the Rheology Advantage™ 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.

Configuring the Upper Heated Plate

The temperature of the upper 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 the Rheology Advantage control module, under the **Options** menu click **Instrument** and then the **Temperature** tab. The window box shown in Figure 7.12 to the right will appear.

A list of the features are described as follows:

- **Cooling Temperature:** The temperature of the circulating water, measured at the inlet: should be entered manually.
- **Cooling range:** This is inversely proportional to the flow rate. Typical values are given in Table 7.2.

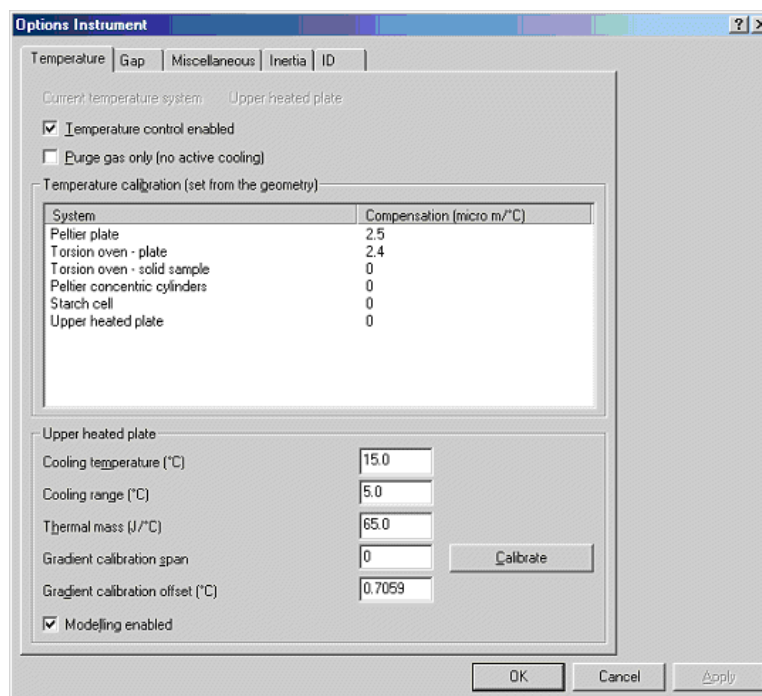


Figure 7.12
UHP Configuration Window

Table 7.2

Feed	Temperature	Flow Rate	Range
Mains tap water	15 °C	0.75 Liter min ⁻¹	5 °C
Fluid circulator	5 °C	0.25 Liter min ⁻¹	15 °C

- **Thermal mass:** The energy required to raise the temperature of the upper platen. It is suggested that the value of 65 J/°C, obtained by TA Instruments, be used unless other information is available.
- **Gradient calibration span:** Arrived at by calibration (see below) although a manual entry may be made.
- **Gradient calibration offset:** Arrived at by calibration (see below) although a manual entry may be made.
- **Modeling enabled:** If this box is checked, the temperature of the upper platen will be matched as closely as possible to that of the Peltier plate during heating or cooling. This means that the heating or cooling rate of both platens is constrained to that of the slower of the two (usually the upper platen). To remove this constraint, allowing the faster platen to change temperature more rapidly than the slower, uncheck this box.

Calibration of the Upper Heated Plate

The temperature of the Upper Heated Plate is read from a probe positioned within the Upper Heated Plate heat spreader 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 Rheology Advantage is that of the Peltier probe. For best performance the Upper Heated Plate probe should be calibrated to the temperature of the upper geometry plate.

NOTE: Calibration should 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

NOTE: Le calibrage devrait être effectué sur l'installation de l'Upper Heated Plate, et au moins annuellement ensuite. La routine de calibrage peut prendre plusieurs heures, et il est plus efficace d'effectuer un calibrage simple avec plus de points, plutôt que plusieurs calibrages avec peu de points.

During the automatic calibration routine a heat flow sensor is used to determine 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 Upper Heated Plate 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 Upper Heated Plate probe to obtain the appropriate offset and span values.

1. Under the **Options** menu click **Instrument** and then the **Temperature** tab. The window, shown in Figure 7.12 on the previous page, will be displayed.
2. Ensure that the **Cooling temperature** and **Cooling range** boxes contain the appropriate values.
3. Click **Calibrate**. The **Calibrate Zero Heat Flow** window, shown in Figure 7.13 shown to the right, will be displayed.

The parameters shown on the window are described as follows:

- **Start Temperature:** Temperature at which calibration is to begin.
- **End Temperature:** Temperature at which calibration is to end.
- **Number of Points:** Number of temperature points, which will be at equal intervals.

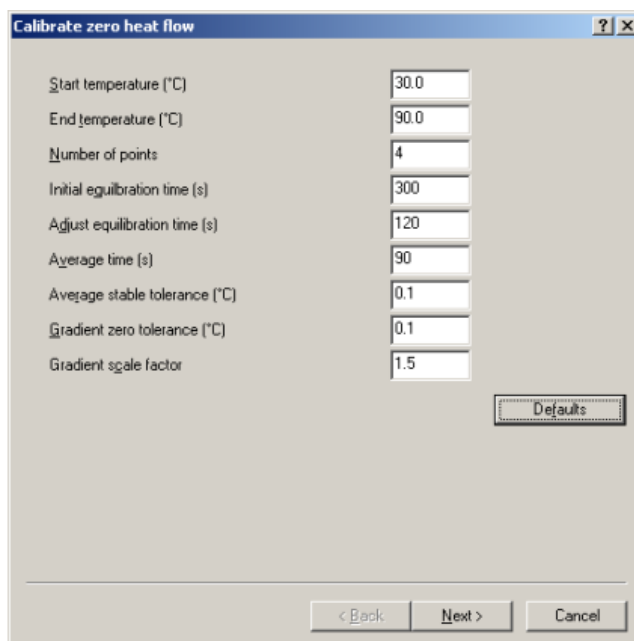


Figure 7.13
UHP Calibrate Zero Heat Flow Window

- **Initial Equilibration Time:** Time at each set temperature, before readings are taken.
- **Adjust Equilibration Time:** Time after each adjustment to the Upper Heated Plate temperature, before readings are taken.
- **Average Time:** Time after each adjustment, over which successive temperature readings are averaged to provide a data point.
- **Average Stable Tolerance:** Range within which two successive data points must fall for the temperature to be accepted as stable.
- **Gradient Zero Tolerance:** Once stability is achieved, the last data point is compared with the set temperature. If the difference is not more than the gradient zero tolerance, the set temperature is accepted as the temperature value. If the difference is greater than the gradient zero tolerance, a further adjustment is made to the Upper Heated Plate temperature.
- **Gradient Scale Factor:** It is suggested that the default value of 1.5 should be used unless other information is available.

4. Click **Next**. A window similar to that shown in Figure 7.14 is displayed. Follow the instructions given. The Upper Heated Plate calibration box and zero value plug are shown in Figure 7.15 below.

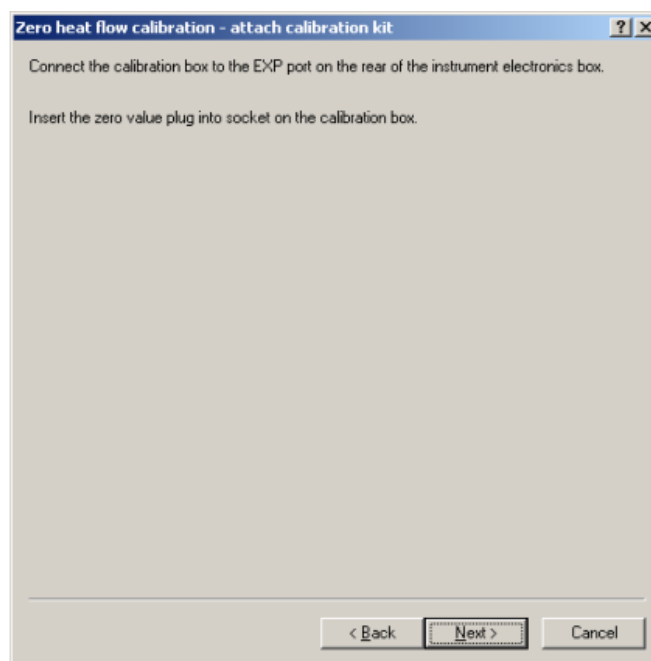


Figure 7.14
Zero Value Determination Instructions

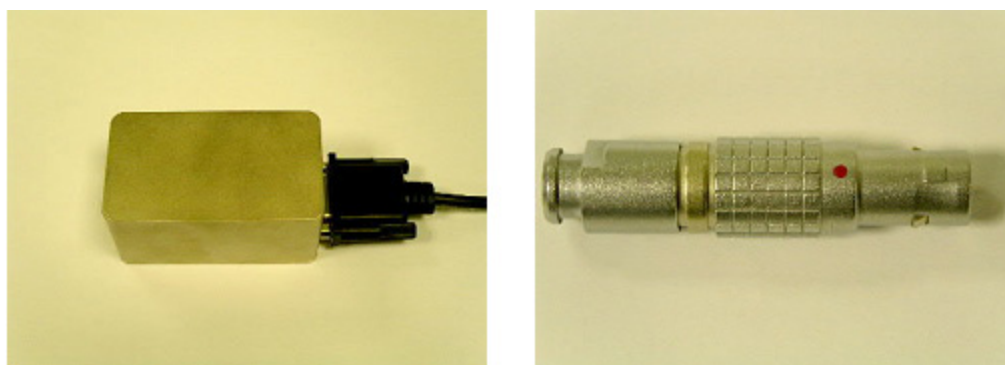


Figure 7.15
Upper Heated Plate Calibration Box (Left) and Zero Value Plug (Right)

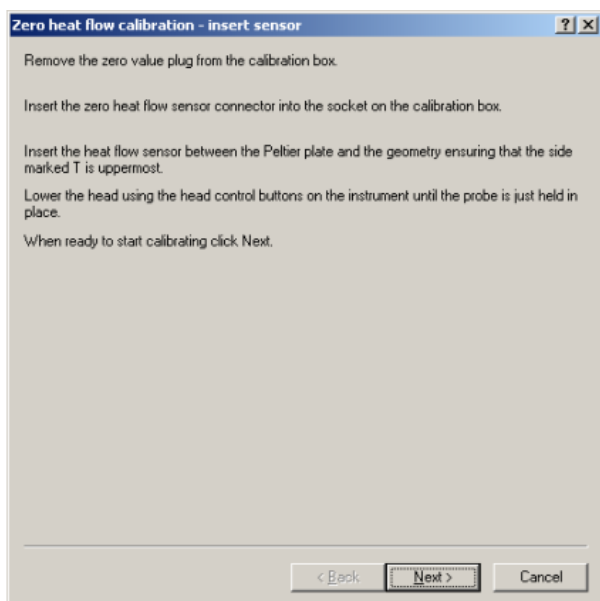


Figure 7.16
UHP Calibration Instructions

5. When the zero value calibration has been completed a window, similar to that shown in Figure 7.16, will be displayed. Follow the instructions given. The zero heat flow sensor is shown connected to the calibration box in Figure 7.17 below.

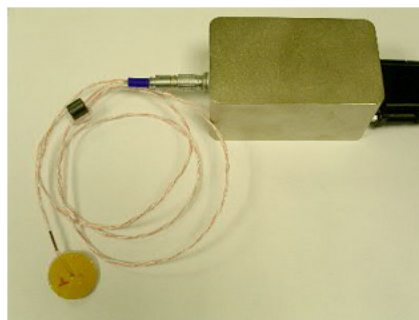


Figure 7.17
Zero Heat Flow Sensor Connected to Calibration Box (Note the T on the upperside of the sensor.)

6. Use the instrument **Head UP** and **Head DOWN** buttons to position the heat flow sensor between the Peltier plate and the geometry as shown in Figure 7.18.

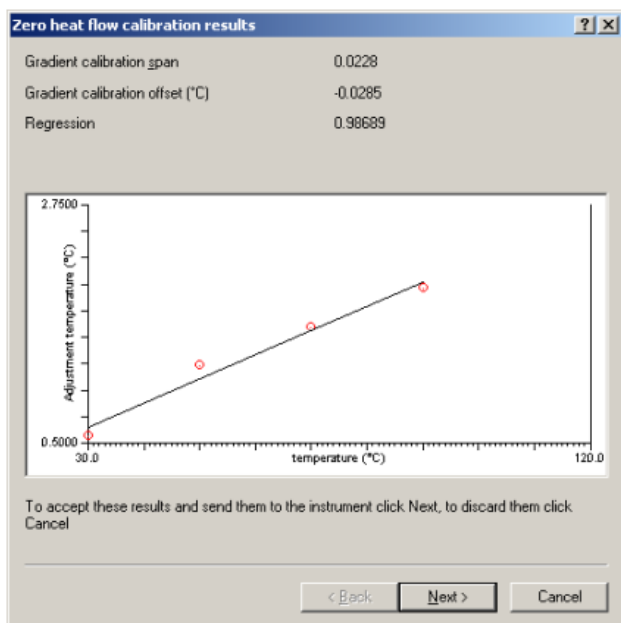


Figure 7.19
Results of UHP Calibration

7. Click **Next** to begin calibrating as instructed (see Figure 7.16, above left). When the calibration is complete the results will be displayed as shown in Figure 7.19 (to the left).

The graph shows the temperature difference between the set temperature and the temperature read by the Upper Heated Plate heat spreader probe, plotted against set temperature.

The **Gradient calibration span** is the slope of the best-fit straight line through the data, and the **Gradient calibration offset** is the intercept.



Figure 7.18
Positioning the Heat Flow Sensor

8. To accept the values click **Next**. The instrument firmware will automatically be updated with these values.

9. When the calibration is finished, raise the instrument head, and remove the calibration sensor. Remove the connector from the electronics box.

NOTE: For safety reasons the temperature control is set to idle at the end of the calibration routine, although the final temperature will still be displayed as the set temperature.

Clamping the Air Bearing

An air-bearing clamp is provided for use with the Upper Heated Plate. Never attach or remove the clamp unless the air supply is connected and switched on.

To attach the clamp

1. Remove the geometry holder from the instrument shaft, and remove the geometry from the holder (see Figure 7.20).
2. Replace the geometry holder
3. Push the clamp up onto the drawrod and attach it by turning the drawrod counterclockwise (anticlockwise).

To remove the clamp hold it firmly and release it by turning the drawrod clockwise.

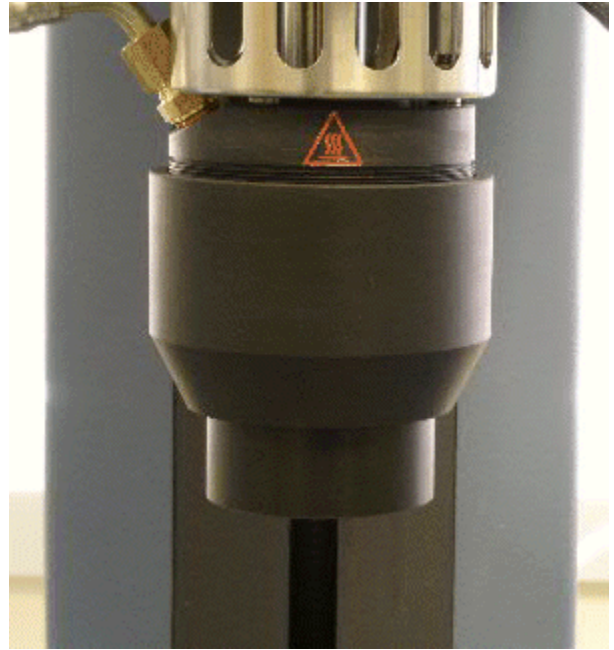


Figure 7.20
Air-Bearing Clamp

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 Liter per minute (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.

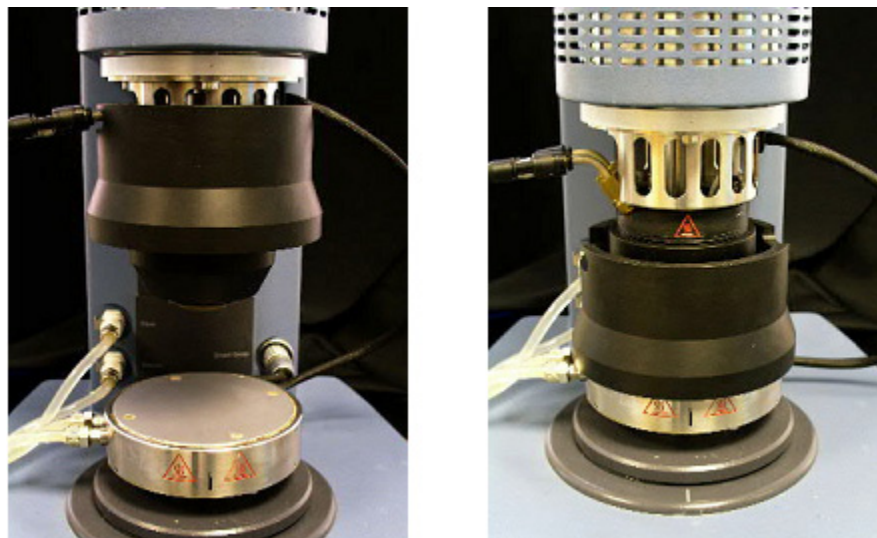


Figure 7.21
Sample Cover
Up Position (Left) and Down Position (Right)

Chapter 8

Do's and Don'ts

Overview

Please read this chapter thoroughly before using your rheometer. It may be helpful to prepare a copy of these points and place them in a prominent position near the instrument.

DO

- ... ensure the air supply is always very clean at a stable pressure. A coarse filter and desiccant dryer system and double filter regulator assembly should be used.
- ... look out for water collection in the filter bowls before and during use of the instrument. Drain the filter bowls whenever necessary. (See Chapter 5.)
- ... look for visible signs of dirt on the filter elements in the filter regulator before use. Replace whenever necessary.
- ... replace the air-bearing clamp when the air is ON, and always ensure it is in place when the instrument is moved or when the air is switched off.
- ... refit the air connector plug supplied with the instrument whenever the air line is disconnected.
- ... disconnect and blast air through the air line whenever starting up after any period which air has been switched off. (Not necessary every morning, unless you know your air supply tends to accumulate water overnight.)
- ... connect and switch on air supply before switching the instrument on.
- ... switch on water supply to the Peltier plate before switching the instrument on.
- ... use good laboratory practice when using the instrument. Wear safety glasses and protective clothing where necessary.

DON'T

- ... operate the instrument without the correct air supply.
- ... remove the air connector plug until ready to attach purged air supply.
- ... unnecessarily touch the air-bearing spindle unless the air is on. This includes attaching and removing geometries.
- ... use the rheometer head as a lifting point.
- ... operate the instrument without a water supply if you have a Peltier System.
- ... disconnect or connect any cables, leads etc. while the power is on.
- ... be frightened of using the instrument for the first time.

Appendix A

Useful Information

Moments of Inertia

The inertia of each geometry can be measured directly via TA Instruments rheology software. Below is a table that shows the approximate inertia values for cone and plate and parallel plate systems. You are advised to use the automatic inertia measurement feature in the software. (See the online help for information).

Units $\mu\text{Nm s}^2$.

	Stainless Steel		Acrylic	
Diameter (mm)	Standard	Solvent Trap	Standard	Solvent Trap
20	1.06	2.80	0.45	0.43
40	4.35	6.92	1.39	1.34
60	17.70	23.32	4.77	3.03

Calculations for Moments of Inertia

Sometimes it may be necessary for you to manually calculate the moment of inertia. Below are the relevant equations you will need.

Cone

Axis through vertex and center of circular base.

$$inertia = \frac{1}{10} \rho r^5$$

r radius

α angle of cone (degrees)

ρ density

Cylinder

Axis through center of cylinder.

$$inertia = \frac{1}{2} h r^4$$

h height of cylinder

The approximate densities ($\times 10^9 \mu\text{Nm}^{-4}\text{s}^2$) are:

Steel	7.83
Acrylic	1.19
Aluminium	2.71

Appendix B

Symbols and Units

The following symbols are used throughout. The instrument can be used with either cgs or SI units depending upon the preference.

<i>Measurement</i>	<i>CGS Unit</i>	<i>Symbol</i>	<i>SI Unit</i>	<i>Symbol</i>	<i>Conversion from CGS to SI</i>
Area	square centimeters	cm ²	square meters	m ²	10 ⁴ cm ² = 1m ²
Force	dyne	dyne	Newton	N	10 ⁵ dyne = 1N
Length	centimeters	cm	meter	m	10 ² cm = 1m
Mass	gram	g	kilogram	kg	1000g = 1kg
Plane Angle	Radian	rad	Radian	rad	No change (360°=2π rad)
Temperature	degree Celsius (Centigrade)	°C	Celsius	°C	No change
Time	second	s	second	s	No change
Volume	cubic	cm ³	cubic	m ³	10 ⁶ cm ³ = 1m ³

<i>Parameter</i>	<i>Symbol</i>	<i>CGS Unit</i>	<i>SI Unit</i>	<i>Conversion CGS to SI</i>
Angular Displacement	ω	rad	rad	No change
Angular Velocity	ω	rad s ⁻¹	rad s ⁻¹	No change
Compliance	J	cm ² dynes ⁻¹	m ² N ⁻¹	x 10
Cone Angle	α	degrees	degrees	No change
Elastic Shear Modulus		dyne cm ⁻² rad ⁻¹	N m ⁻² rad ⁻¹	x 10 ⁻¹
Shear Rate	$\dot{\gamma}$	s ⁻¹	s ⁻¹	No change
Shear Strain	γ			No change
Shear Stress	σ	dyne cm ⁻²	N m ⁻² (Pa)	x 10 ⁻¹
Torsional Force	τ	dyne cm	N m	x 10 ⁻⁷
Viscosity	η	Poise (P)	Pa.s	1 cP = 1 mPa.s

Appendix C

Geometry Form Factors

Cone/Plates

Dimensions				Form Factors		
Angle (°)	Diam. (mm)	Truncation (approx.) (µm)	Sample volume (ml)	Shear rate	Shear stress	Viscos. (Pa.s)
0	20					
0.5	20	13	0.03	114.6	0.4774	0.00416
1.0	20	26	0.06	57.3	0.4774	0.00833
1.5	20	39	0.09	38.16	0.4774	0.01251
2.0	20	52	0.12	28.65	0.4774	0.01666
4.0	20	105	0.24	14.33	0.4774	0.03331
0.0	40					
0.5	40	13	0.15	114.6	0.0596	0.00052
1.0	40	26	0.30	57.3	0.0596	0.00104
1.5	40	39	0.45	38.16	0.0596	0.00156
2.0	40	52	0.60	28.65	0.0596	0.00208
4.0	40	105	1.20	14.33	0.0596	0.00416
0.0	60					
0.5	60	13	0.60	114.6	0.0177	0.00015
1.0	60	26	1.20	57.3	0.0177	0.00031
1.5	60	39	1.80	38.16	0.0177	0.00046
2.0	60	52	2.4	28.65	0.0177	0.00618
4.0	60	105	4.8	14.33	0.0177	0.00124

Concentric Cylinder Dimensions

Rotor Type	Rotor Radius R1 (mm)	Stator Radius R2 (mm)	Immersed Height (mm)	Gap (microns)
DIN (conical)	14	15	42	5920
Recessed	14	15	42	4000
Vaned	14	15	42	4000

Rotor Type	Stator Outer Radius R1 (mm)	Rotor Inner Radius R2 (mm)	Rotor Outer Radius R3 (mm)	Immersed Height (mm)	Gap (microns)
Double Gap	20	20.38	21.96	59.5	500

Appendix D

LCD Display Messages

The LCD display on the front of the rheometer electronics box displays useful information and error messages.

Power On Messages

Immediately after power up of the rheometer, the display will show '**Initialising...**' After a few seconds this will change to '**AR2000 7.xx xx/xx/xx**' (x is version dependant). If it does not, there is a problem with the rheometer.

After a few more seconds, the display will either show '**System test Ok**' or '**System test failed Y,**' where Y is an error code as show below:

1	ROM checksum error	Either bad firmware or or hardware fault	Try reloading firmware, otherwise call for service.
2	RAM error	Hardware fault	Call for service.
4	Dual port RAM error	Hardware fault	Call for service.
40	Battery failure	Low battery	Sometimes seen the first time the system is restarted after a firmware upgrade. Cycles power off then on.
80	Backup RAM error	Hardware fault	Call for service.
400	Parameter block checksum error	Either corrupted internal system parameters or hardware fault	Call for service.

Initialising ...

During start up of the rheometer, the following items are shown as initializing:

- Electronics
- Power board
- Instrument

Bearing overspeed

Shown when the rheometer bearing rotation exceeds the specified maximum speed.

Bearing pressure too low

This is displayed if the air supply has been inadvertently switched off while the rheometer was on or if the supply pressure has dropped below the minimum operating pressure.

Encoder index not found

This message can be displayed if the air bearing is not free to move. This can occur if the air-bearing clamp is still attached or if the air bearing lock is on. Failing this, there may be a fault on the position encoder.

Nf gauge fault

This message is displayed if an excessive normal force was detected when attempting to zero the normal force reading. This could be caused by either an excessive force being applied by air bearing/sample/mechanical interference with ETC or a gauge fault.

Nf temp sensor fault

This message is displayed if there are either faulty normal force temp sensors or, if you have for instance, frozen the instrument with excessive liquid nitrogen.

Operator stop event

This message is displayed when the Stop button was pressed while the Rheology Advantage software had the keypad locked. This usually indicates that a test run was in progress and the operator aborted it using the rheometer stop button.

Power cable fault

This indicates that the Power cable may not be plugged in firmly. See Chapter 5 for connection information.

Signal cable fault

This message indicates that the signal cable may not be plugged in firmly. See Chapter 5 for connection information.

Temp sys element fault

This message is displayed because the Peltier or heater element has developed a fault.

Temp system environment

There is a configuration problem with the installed temperature control module. *I.e.*, no water to the Peltier etc. Re-read the manual to check you have set everything up correctly.

Temp system sensor fault

This message is likely caused by fault/damage to the Pt100 or thermocouple.

Other Messages

Other error messages may be displayed. These are usually indicative of problems with the rheometer that can only be fixed by a qualified TA Instruments representative.

Appendix E

TA Instruments ETC Kits

ETC Torsion Rectangular Kit (543307.901)

Item No.	Description	Quantity
1	Lower Clamp Assembly	1
2	Upper shaft	1
3	Clamp set rectangular	1
4	Torque wrench	1
5	Digital Callipers	1
6	Tommy Bar	1
7	0.25 mm spacers	2
8	0.5 mm spacers	2
9	0.75 mm spacers	2
10	Compliance Standard	1
11	ABS polymer samples	10

ETC Parallel Plate Kit (543306.901)

Item No.	Description	Quantity
1	Lower Plate Assembly	1
2	Upper Shaft	1
3	25 mm Parallel plates	1 set
4	40 mm Parallel plates	1 set
5	Melt Ring	10
6	Tommy Bar	1
7	Brass brush	1
8	Brass scraper	1

ETC Disp. Parallel Plate Kit (543308.901)

Item No.	Description	Quantity
1	Lower Plate Assembly	1
2	Upper Shaft	1
3	25 mm Parallel disposable plates	10 set
4	Tommy Bar	1
5	Hex key	
6	Spare clamping screws	

A

air bearing 23 to 24

air-bearing clamp 33
removing 61
Upper Heated Plate (UHP) 77

angles
cones 58

AR 2000 Rheometer
additional precautions 45
air bearing 23
changing the cup 40
checking system 50
components 21
connecting the purge gas 43
connecting to electronics control box 35
cooling 47
description 9
dimensions 29
do's and don'ts 81
electronics 21
error codes 89
flow rate 26
geometries 53
installation requirements 34
internal fuse 34
keypad 49
LCD display messages 89
lifting and carrying 15
location requirements 34
normal force transducer 27
preparing for installation 33
preventing slippage at sample/geometry interface 60
removing packaging 33
repair 18
schematics 20
technical descriptions 23
technical specifications 29, 30
temperature control 26
thermocouple 46
using the stress and shear rate factors 57
water bath 26

Auto GapSet 25

auto-zero gap 49

B

battery
lithium 9

C

cables
connecting 37

calibration 51
Upper Heated Plate (UHP) 74

chemical safety 17

cleaning
filter regulator assembly 52

computer
connecting to electronics control box 35

concentric cylinder systems 56
conical end 56
double concentric 57
form factors 87
recessed end 56
setting up 39

cone and plate system 54, 58
angles 58
material 59
moments of inertia 83

cone and plate systems
form factors 87

controlled-stress technique 20

cooling
controlled 47

Cooling Control Unit (CCU) 65

cross threading 48

cross-hatched geometries 60

cryogenic pressure vessel 47

cup
changing 40

D

deformation 20

desiccant dryer system 81

diameters
cones 58

draw rod 33, 53

drive motor 21

E

edge effects 63

electrical safety 15

electronic control box 47

EMC Conformité 14

EMC Conformity 13

encoder disc 21

Environmental Test Chamber (ETC)
specifications 30

Environmental Testing Chamber. *See* ETC. *See also* ETC

Environmental Testing Chamber (ETC)
installing 41
installing the low temperature accessory 44
internal components 10
kits 93
oven 41
setting up 41

equilibrium time 47

error codes 89

EVENT socket 71

F

filter regulator assembly

cleaning 52

signs of dirt 81

flow rate 26, 44

fluid cables

connecting 37

fluids

used with the Upper Heated Plate (UHP) 70

fuse 34

G

gap

closing 25

overfilling 63

setting 55

underfilling 63

zeroing 25

gap zeroing 25

geometries 45

geometry. *See also* measuring system

aluminum 53

attaching 62

choosing the best 58

concentric cylinders 56

cone and plate 54

connecting for Upper Heated Plate 72

cross-hatched 60

description 53

disconnecting for Upper Heated Plate 72

form factors 87

inertia 83

materials 53 to 58

parallel plate 55

plastic 54

solvent trap version 60

stainless steel 53

I

inert gas 78

installation
requirements 34

L

lab air 34

l'azote liquide 16

LCD display messages 89

levelling 50

lifting and carrying 15

liquid nitrogen 10
handling 17
safety 16

lithium battery 9

location 34

low temperature accessory 44

M

mapping procedure 47

measurements
preventing evaporation 60

measuring system
general description 53

microprocessor 24

moments of inertia
calculation 83 to 84

motor spindle 53

N

- Newtonian viscosity 51
- normal force transducer 27
- Notes, Cautions, and Warnings 8

O

- operation
 - general guidelines 48
 - rheometer 47
- optical encoder 24

P

- packaging
 - removing 33
- parallel plate system
 - 55
 - moments of inertia 83
- parallel plate systems 58
- parallel plates
 - 45
 - cleaning 46
- Peltier Concentric Cylinder System
 - specifications 31
- Peltier Plate
 - 26 to 27
 - installing 37
 - removing 38
- Peltier plate 53
- Peltier Plate System
 - specifications 30
- Peltier plate system 81
- polymers 47
- power cables
 - 10
 - connecting 37

power outlet 34

pressure 44

purge gas
connecting to rheometer 43

R

relative humidity 34

rheology
definition 20

rheometers
brief history 19

Rotational Mapping 24, 24 to 25

Rotor 53

S

safety
chemical 17
electrical 15
EMC Conformity 13
warnings 9

sample
loading 63

sample cover 79

samples
preventing evaporation 60

shear rate 57

shear stress 57

shut down 51

Sir Isaac Newton 19

Smart Swap™ 26
using 37

- Smart Swap Peltier plate 65
- solvents
 - preventing evaporation 60
- specifications 29
 - AR 2000 29
 - ETC 30
 - Peltier concentric cylinder system 31
 - Peltier plate system 30
- Stator 53
- strain 57
- symbols 85
- system check 50

T

- thermal compensation 25 to 27
- thermocouple 46

U

- units 85
- Upper Heated Plate (UHP)
 - air-bearing clamp 77
 - attaching to rheometer 66
 - calibrating 74
 - circulating fluids 70
 - configuring 73
 - connecting the Cooling Control Unit (CCU) 69
 - connection configurations 68
 - cooling water 68
 - description 65
 - gas atmosphere 65, 78
 - geometry holder
 - disconnecting 72
 - introduction 65
 - modeling enabled 73
 - Pt100 65
 - sample cover 79
 - using inert gas atmosphere 78
 - zero heat flow sensor 76
 - zero value plug 75

V

viscosity
definition 19

W

warnings
important information 9

water
connecting to rheometer 36

water bath 26

work surface 34

Z

Zero Gap 49

zero heat flow sensor
Upper Heated Plate (UHP) 76

