AR 2000 Rheometer

Rheometrics Series Operator's Manual

PN 500106.002 Rev. L
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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.

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 recommendé 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'altérerons 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 a température ambiante en frottant légèrement avec un chiffon imbibé de solvant.

Les cables externes doivent être toujours separés du cable d'alimentation. S'en assurer à chaque installation. De même, tout cable 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 reservoir 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 securité.

Utiliser l'azote liquide avec précautions car une utilisation inadéquate peut provoquer des suffocations. Stocker et utiliser dans une pièce suffisament ventilée. Ne pas pénétrer dans une pièce remplie d'azote avant d'en avoir evacué 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 securité 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 securité 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	
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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 Smart Swap™ 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 etant sous hautes tensions, l'entretien et la réparation des pièces internes doivent être effectues 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 extrèmement chaudes ou froides peuvent être exposées. Prendre toutes précautions necessaires 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 chassis 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 sectiion 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.

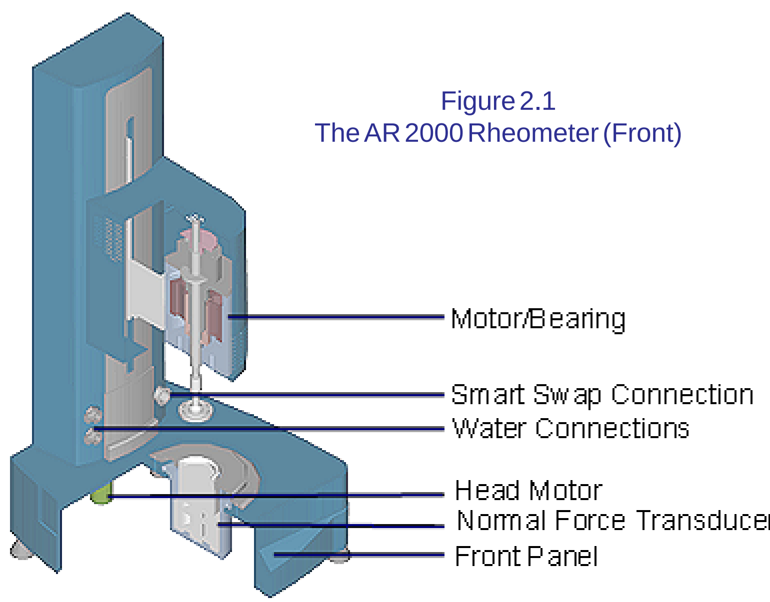


Figure 2.1
The AR 2000 Rheometer (Front)

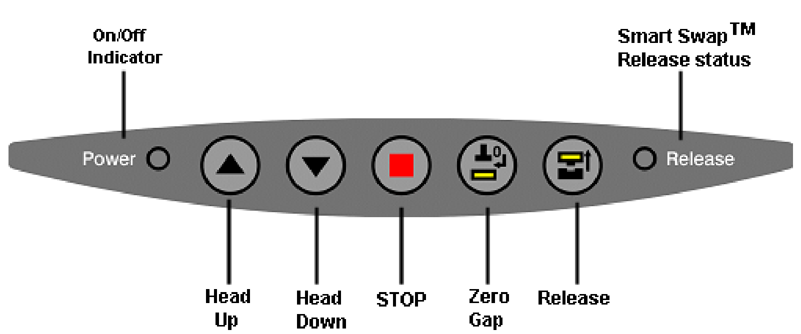


Figure 2.2
The AR 2000 Rheometer (Control Panel)

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.

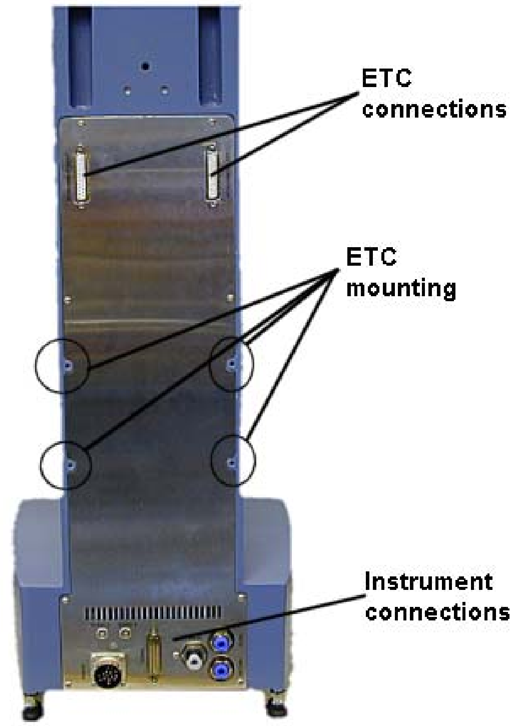


Figure 2.3
The AR 2000 Rheometer (Rear)

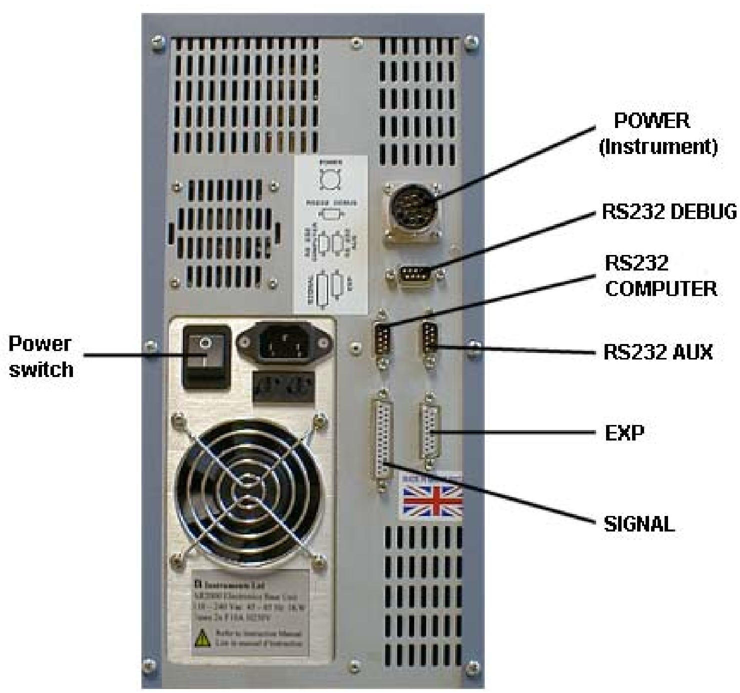


Figure 2.4
Electronics Control Box (Rear)

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 (<10µm) 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 1µm, providing an extremely smooth finish.

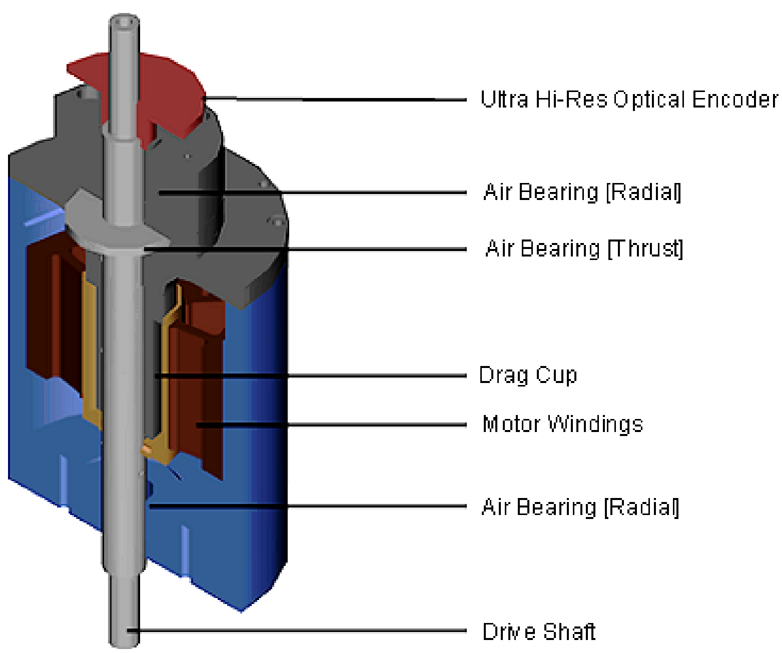


Figure 3.1
The Rheometer Head

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 $0.5\ \mu m°C^{-1}$. 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.

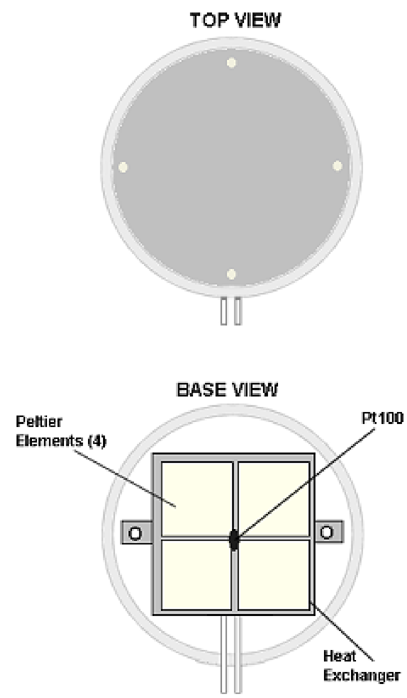


Figure 3.2
The Peltier Plate

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.

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 µNm to 200 mNm
Frequency Range	0.12 µHz 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	-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
Typical Ramp Rate	30 °C min^{-1}
Ramp Rate (20 to 100 °C)	50 °C min^{-1}
(100 to 150 °C)	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	50°C to 600°C
LN2 cooling	-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 vortex air cooler	 20°C to 150° -30°C to 55°C -5°C to 150°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.

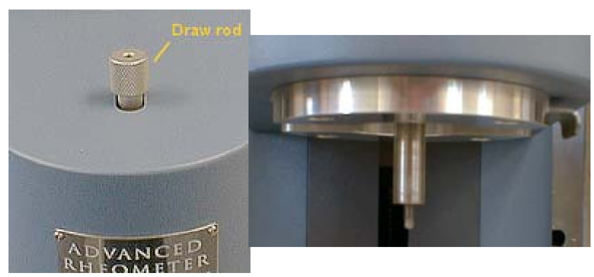


Figure 5.1
Inserting the Draw Rod

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

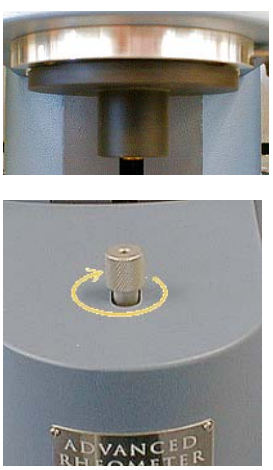


Figure 5.2
Performing Step 3

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.

Models with Air Bearing Lock

If the AR 2000 you own has an air bearing lock, follow these steps:

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

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°C ±4°C, relative humidity 50 ±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 ±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

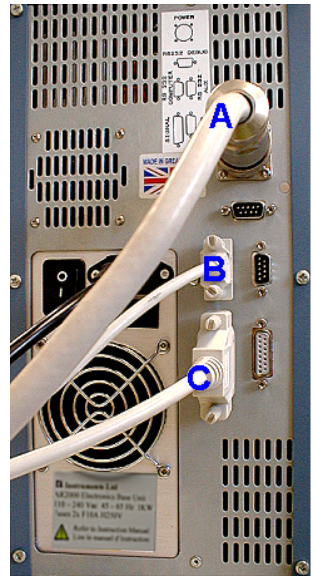
Connecting the system together should present no problems, as long as you use instructions found in the following sections.

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 5.3).

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).



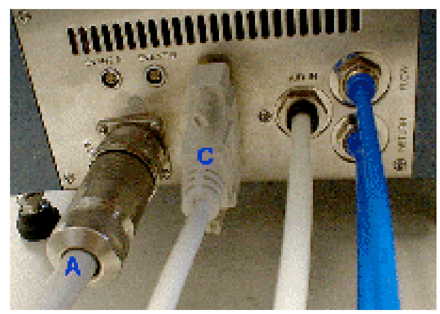


Figure 5.3
Cable Connections

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 5.3).

2. Push the 9-pin male connector into the serial port socket on the back of the socket on the computer.

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 5.4. A continuous green light indicates that the attachment can be fitted.

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

    Le dévérouillage restera seulement actif pendant 10 secondes.

2. Fit the attachment as shown in Figure 5.5 below, ensuring it is aligned correctly.

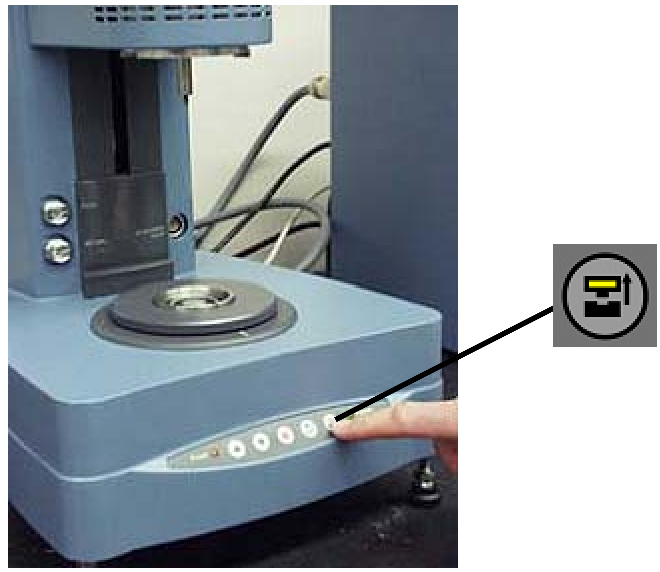


Figure 5.4
Press the Release Button

Figure 5.5
Fitting the Attachment

3. Connect the power and fluid cables. See Figure 5.6.

4. When the green status light goes out, the rheometer is ready for use.

Figure 5.6
Connecting Power and Fluid Cables

Removing the Peltier Plate

1. Press the '**Release button**' on the control panel (see Figure 5.7). 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 5.8.

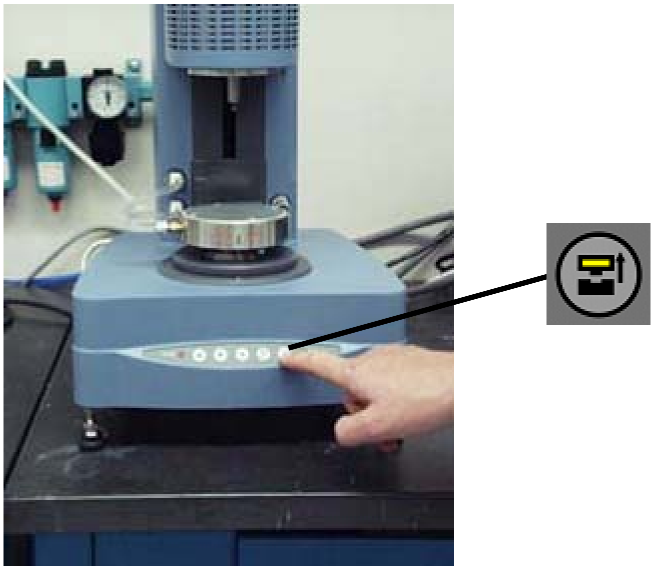


Figure 5.7
Releasing the Attachment

Figure 5.8
Removing the Peltier Plate

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

Le dévérouillage restera actif pendant 10 secondes. A l'issue de ces 10 secondes la plaque sera vérouillée 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 5.9. A continuous green light indicates that the attachment can be fitted.

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

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

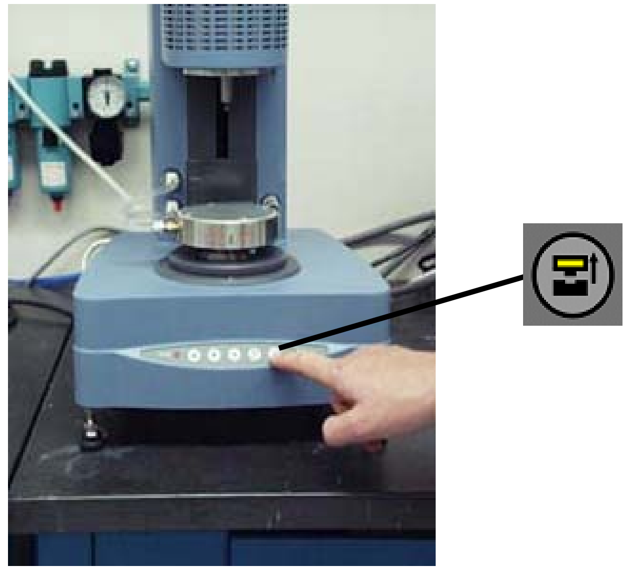


Figure 5.9
Releasing the Attachment

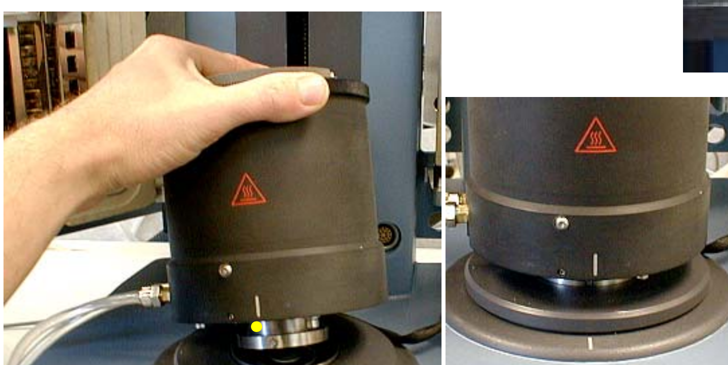


Figure 5.10
Fitting the Cylinder Attachment

4. Connect the power and fluid cables as shown in Figure 5.11 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.

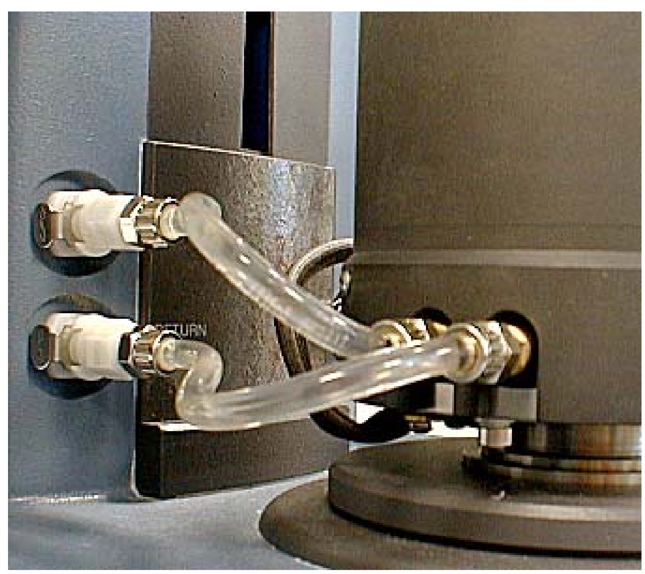


Figure 5.11
Connecting Fluid Cables

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 5.12 to the right. You can now set up the measuring geometry in the rheometer software and set the gap explained in the online help.

Figure 5.12
Lowering Rheometer Head

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.

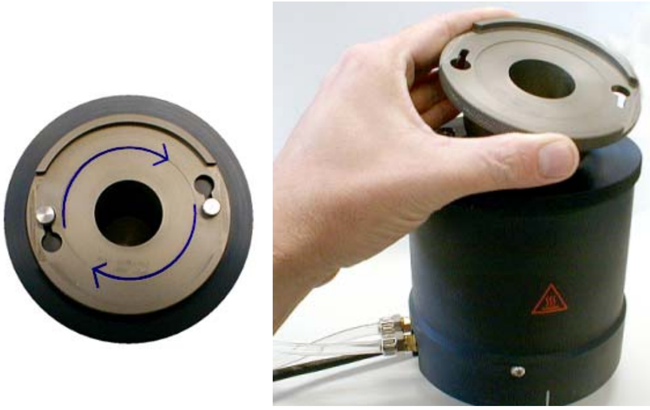


Figure 5.13
Changing the Cup

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 5.14) 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 5.15) and then use the handles on the oven doors to lift it onto the two top screws. Lightly tighten these screws.

Figure 5.14
Mounting Screws

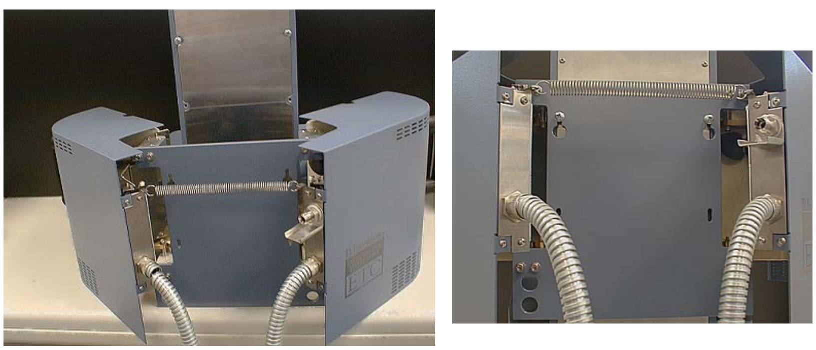


Figure 5.15
The ETC Open

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 5.16.

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

Figure 5.16
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 dévérouillage restera actif pendant 10 secondes.

12. Fit the lower attachment, ensuring it is aligned correctly. See Figure 5.18 below.

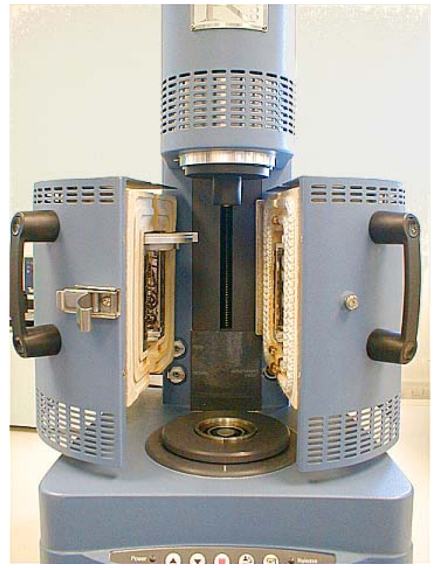


Figure 5.17
ETC with Open Oven Doors

Figure 5.18
Fitting the Lower Attachment

13. Connect the cable from the lower attachment to the rheometer as seen in Figure 5.19.

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.

Figure 5.19
Connecting the Cable from the Lower Attachment to the Rheometer

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.

18. Connect the purge gas to the rheometer as seen in Figure 5.20 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 5.22. Otherwise, connect as shown in Figure 5.21 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.

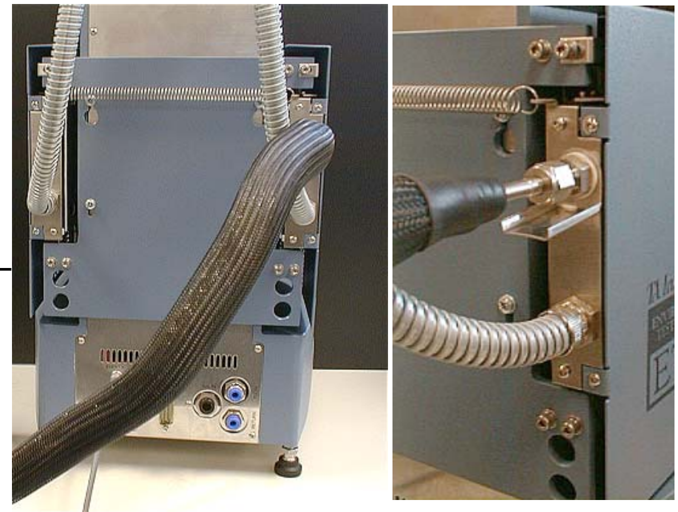


Figure 5.20
Connecting the Purge Gas

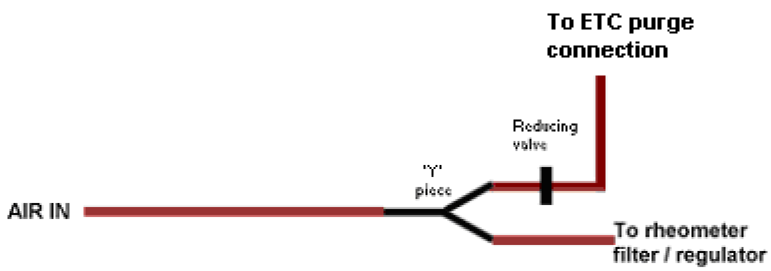


Figure 5.21
ETC Connections Using Air as the Agitation Gas

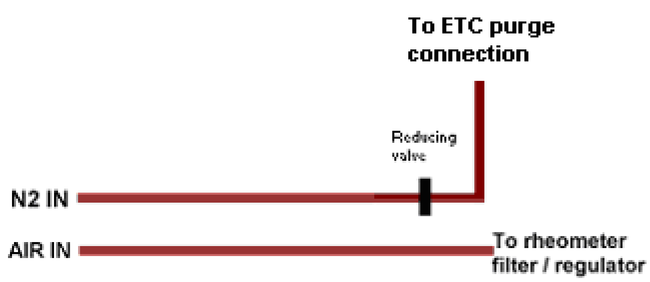


Figure 5.22
ETC Connections Using Nitrogen as the Agitation Gas

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 5.24 and Figure 5.25).

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 5.23. Connect the purge gas from the flow control assembly to the rheometer.

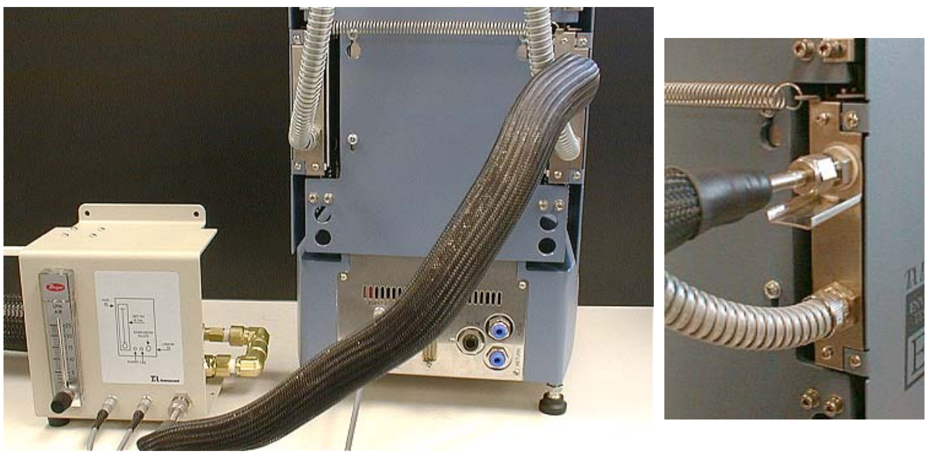


Figure 5.23
Connecting the Hose

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 5.25, otherwise follow Figure 5.24.

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.

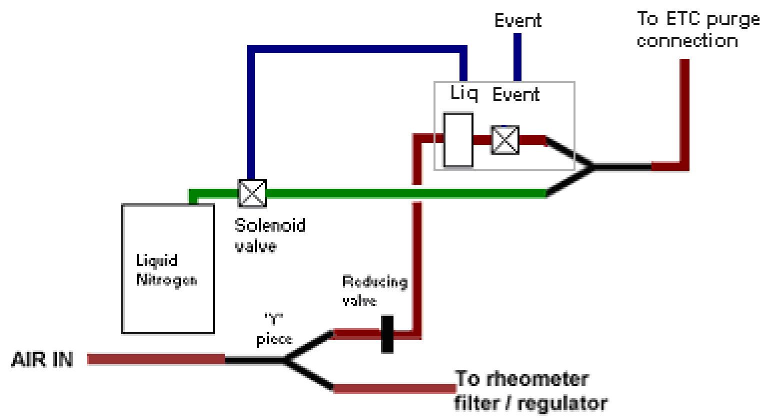


Figure 5.24
ETC Connections with LN2

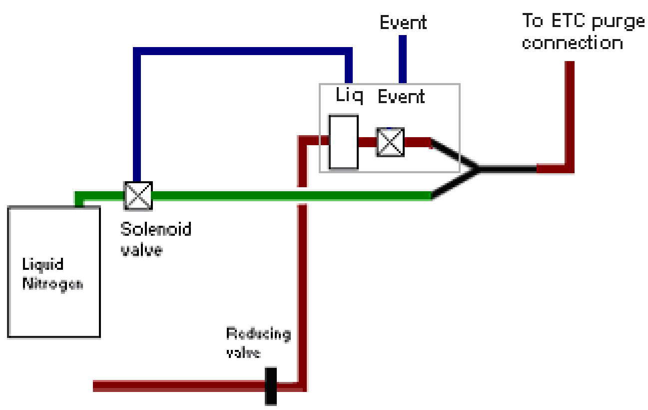


Figure 5.25
ETC Connections with Nitrogen

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.

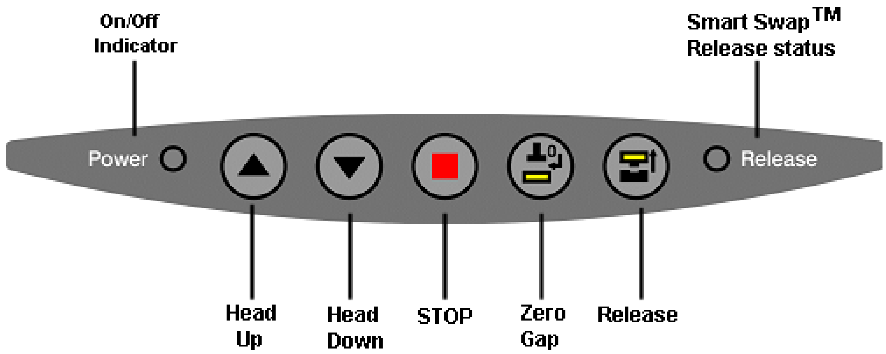


Figure 5.26
AR 2000 Keypad

Feature	Description
On/Off Indicator	A continuous red light indicates that rheometer is receiving power.
Smart Swap Release Status	
Off	Attachment holder is locked.
Flashing green	Power to attachment is removed (holder still locked.)
Continuous green	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 <u>not</u> 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. (Cannon S600 oil, which has a nominal viscosity of 1.4 Pas at 25°C, or PTB 1000A, which has a nominal viscosity of 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 5.27.

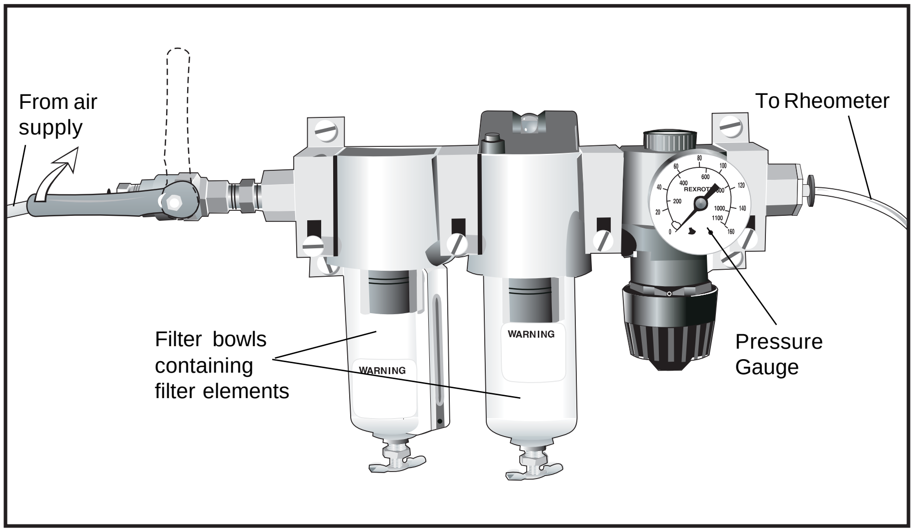


Figure 5.27
The Filter Regulator Assembly

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.

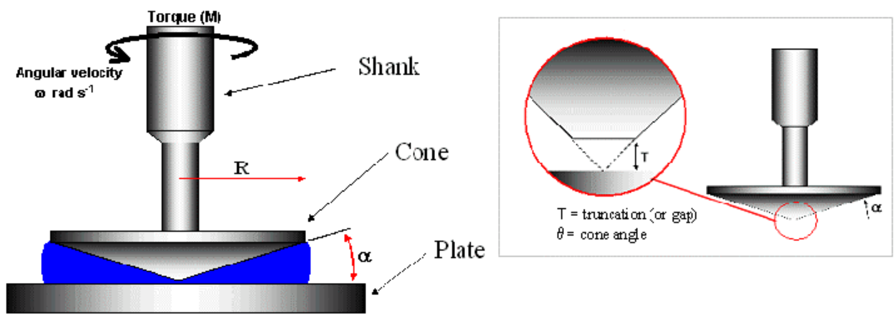


Figure 6.1
The Cone and Plate

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

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

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

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

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

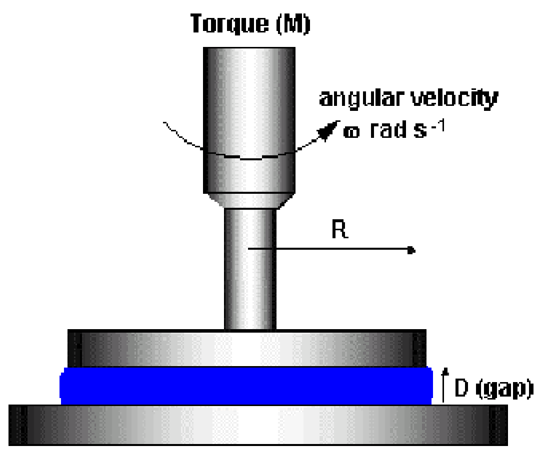
Cone and plate geometries are generally used for single-phase homogeneous samples or samples with submicron particles. Samples containing particulate matter are usually unsuitable for cone and plate geometries as the particles will tend to migrate to the apex of the cone and will get jammed in the truncation 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 mm, you should set the gap to at 1000 mm.
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.



$$\text{Shear rate } (s^{-1}) = F\gamma\,\omega \qquad \text{where } F\gamma = \frac{R}{D}$$

$$\text{Shearstress } (Pa) = F_\sigma\, T \qquad \text{where } F_\sigma = \frac{2}{\pi R^3}$$

Figure 6.2
The Parallel Plate

Concentric Cylinders

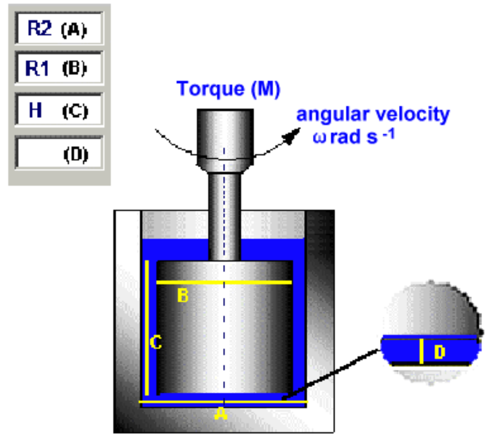
Concentric cylinder systems (or cup and bob) are generally used for lower viscosity samples that may not be held within the gap of cone and plate or parallel plate systems. (See later in this chapter for information on setting up and using the concentric cylinder systems.)

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.

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

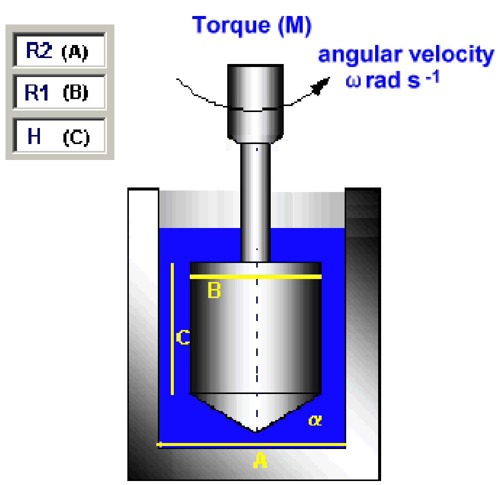
where $F\gamma = \dfrac{R_2^2 + R_1^2}{R_2^2 - R_1^2}$

Shear stress $(Pa) = F_\sigma M$

where $F_\sigma = \dfrac{R_1^2 + R_2^2}{4\pi H R_1^2 R_2^2}$

Recessed End

Figure 6.3
Recessed End



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

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

Conical End

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.

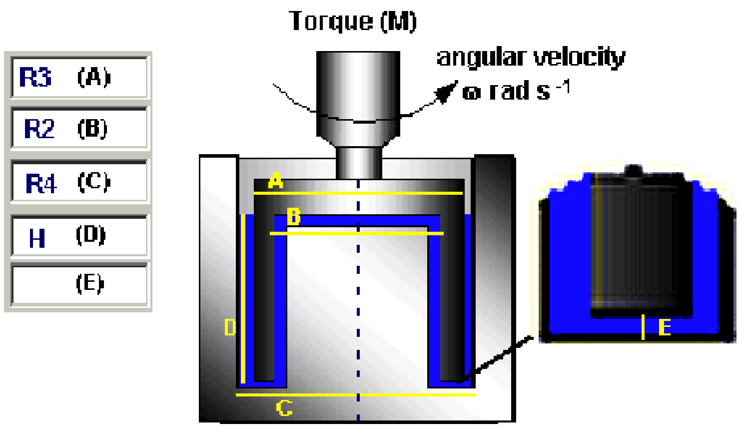


Figure 6.5
Double Concentric

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.

$$Shear\ stress(Pa) = F_\sigma\ M$$

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

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.

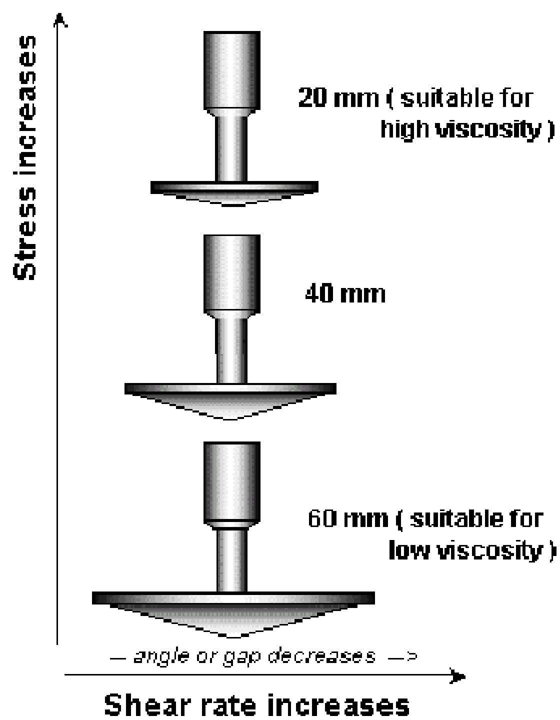


Figure 6.6
Choosing Geometry Angle and Diameter

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.

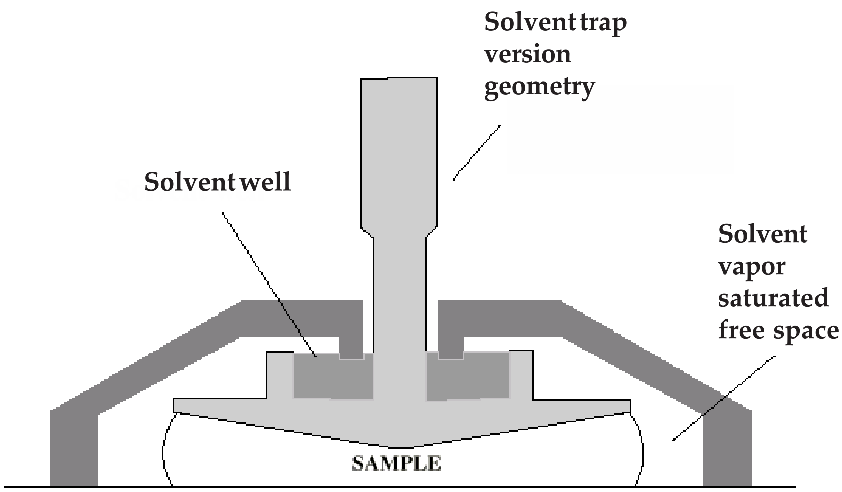


Figure 6.7
Solvent Trap Cover and Geometry

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 counterclock-wise (anticlockwise).

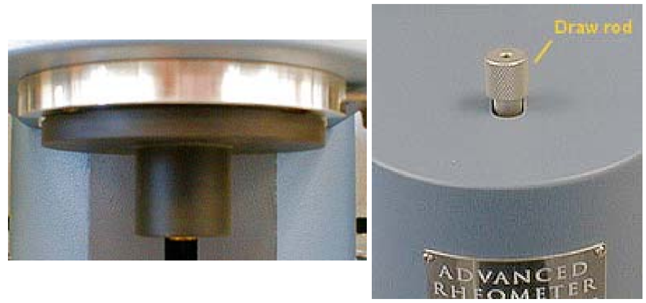


Figure 6.8
Removing the Air Bearing Clamp

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.

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.

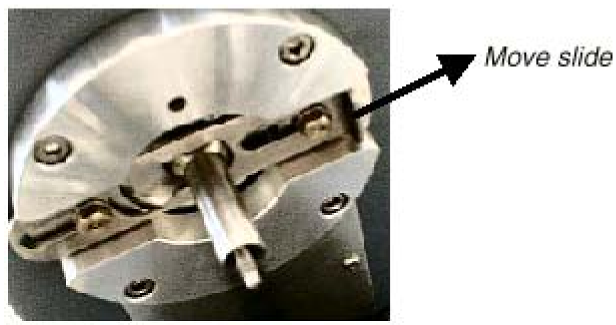


Figure 6.9
Slide Lock Away

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.

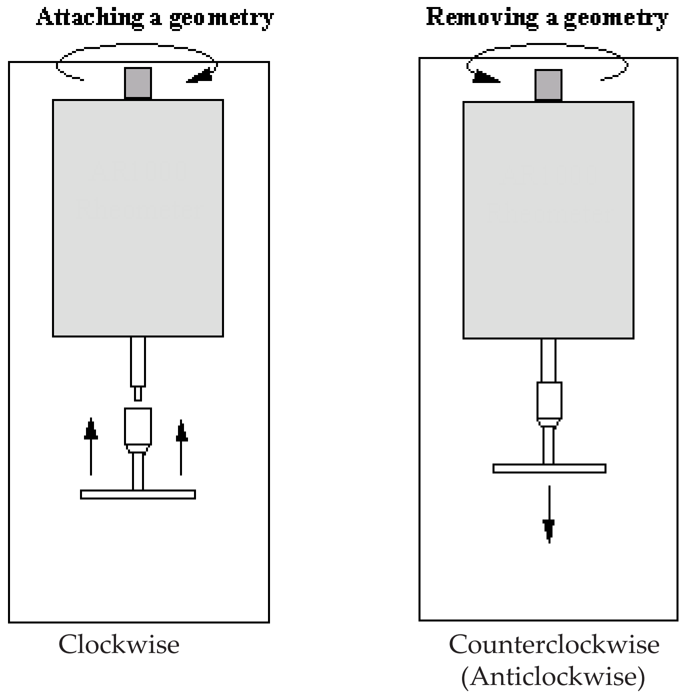


Figure 6.10
Attaching/Removing A Geometry

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.

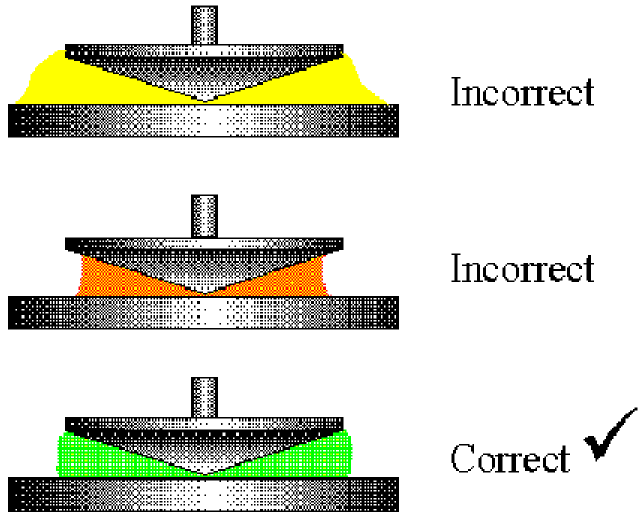


Figure 6.11
Loading the Sample

- 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.

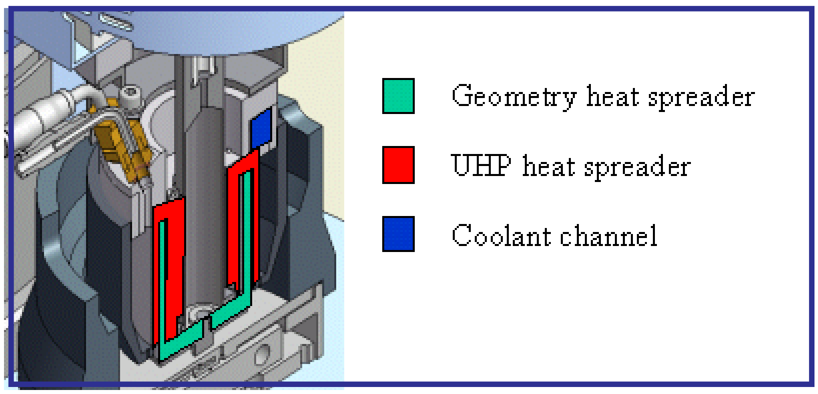


Figure 7.1
Exploded View of the UHP and Upper Geometry Holder

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 vortex air, water, or other fluid carried in the coolant channel.

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

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

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

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).

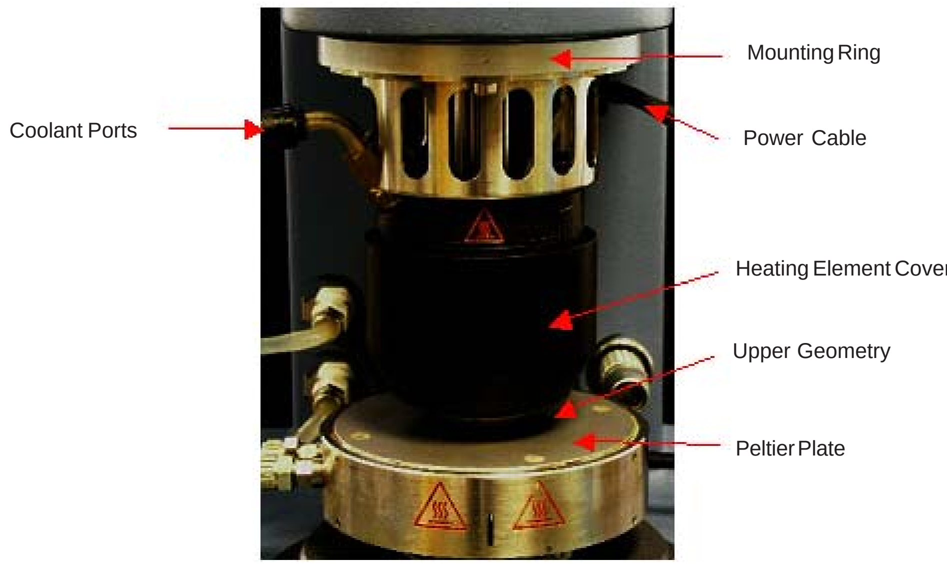


Figure 7.2
The Upper Heated Plate Shown Mounted on an AR 2000 Rheometer

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).

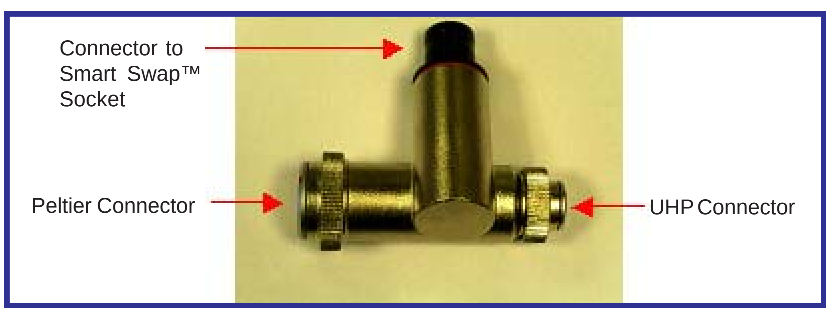


Figure 7.3
The Smart Swap™ UHP Adaptor

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

Installing the (Optional) Vortex Air Cooler

Follow the steps below to attach and connect the vortex air cooler to the AR 2000. Refer to the figures as needed.

1. Use the two screws provided to mount the vortex air cooler bracket to the rear of the AR 2000 casting as shown in Figure 7.5 to the right.

2. Clip the vortex air cooler into the spring clips with the brass muffler extending upward as shown in Figure 7.6 below.

Figure 7.5
Mounting the Bracket

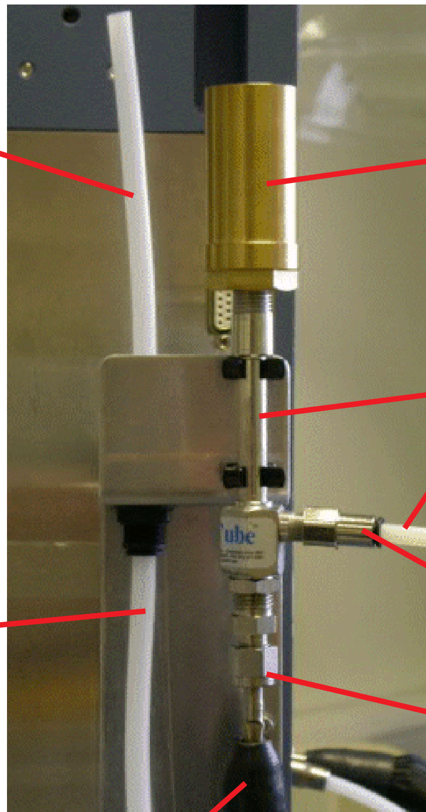


Figure 7.6
Attaching the Vortex Air Cooler

3. Remove the metal push-fit connector from the inlet port on the UHP and fit the Swagelok adapter supplied in the kit. See Figure 7.7 to the right. (Note that once this has been fitted, it cannot be removed. Returning to the push-fit connector will require the supplied adapter.)

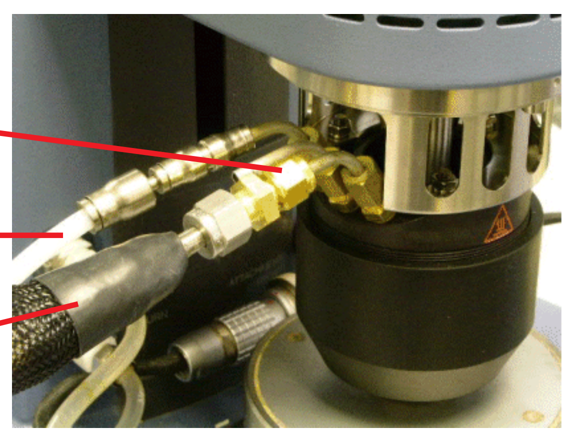


Figure 7.7
UHP Swagelok Adapter

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

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

6. Cut 150 mm of white 6-mm O.D. tubing and connect one end to the upper bulkhead fitting on the vortex air cooler bracket. The other end is left open to vent to the atmosphere.

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

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

9. Connect the event socket on the valve bracket to the EVENT B socket on the rear of the AR 2000 rheometer using the cable provided.

Table 7.1

	Minimum Temperature	Maximum Temperature
Vortex Air Cooler	–5 °C	150 °C

NOTE: If you find a reduction in the expected cooling performance, check that there is exhaust air flowing from the white 8-mm O.D. tubing. If there is limited or no air flow, this is an indication that the cold end of the vortex tube is blocked with ice, formed by condensing moisture in the air supply. The tube can be taken apart and ice removed, but the only long-term solution is to supply drier air.

Configurations for the Cooling Water

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

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

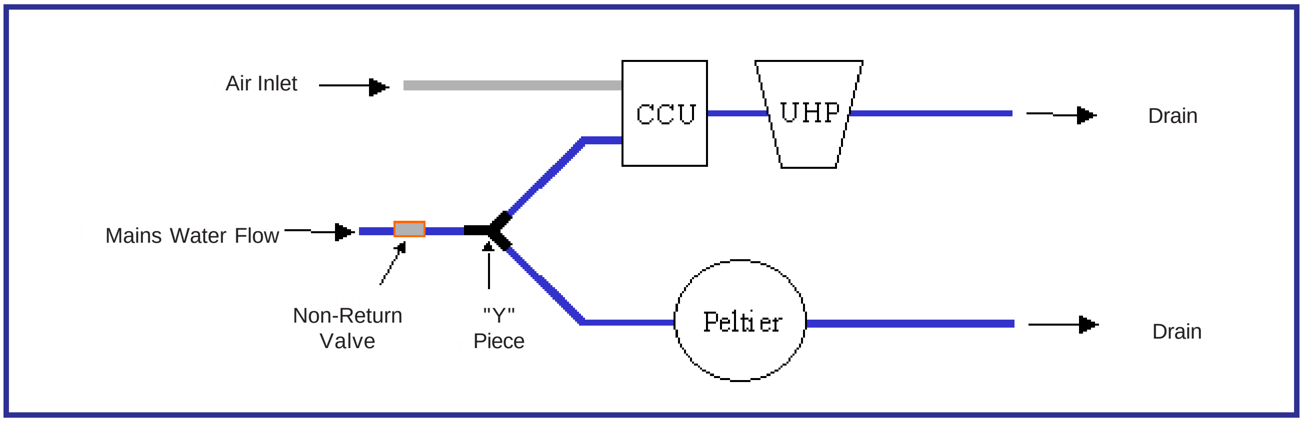


Figure 7.8
Cooling Water Configuration 1
Mains Water Supplying Both Peltier and Upper Heated Plate

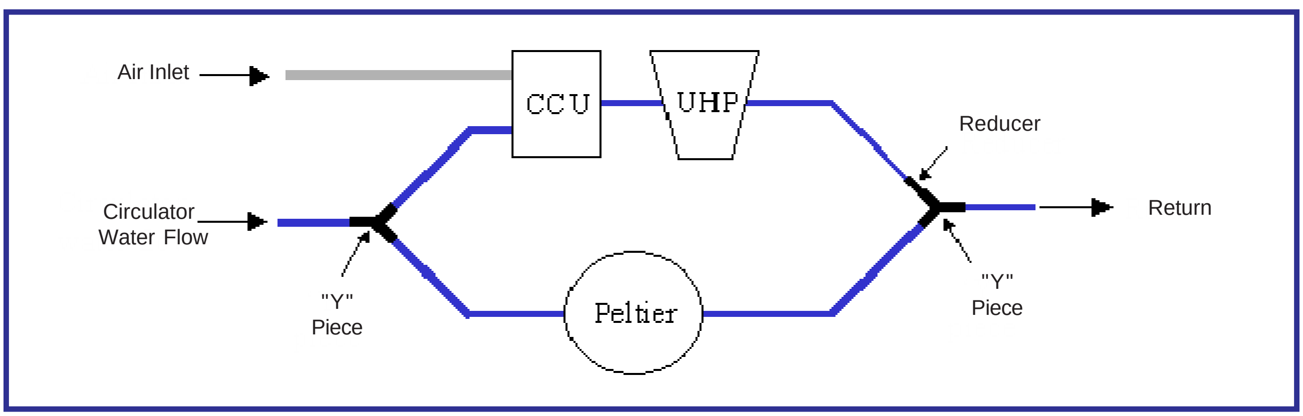


Figure 7.9
Cooling Water Configuration 2
Fluid Circulator Supplying Both Peltier and Upper Heated Plate

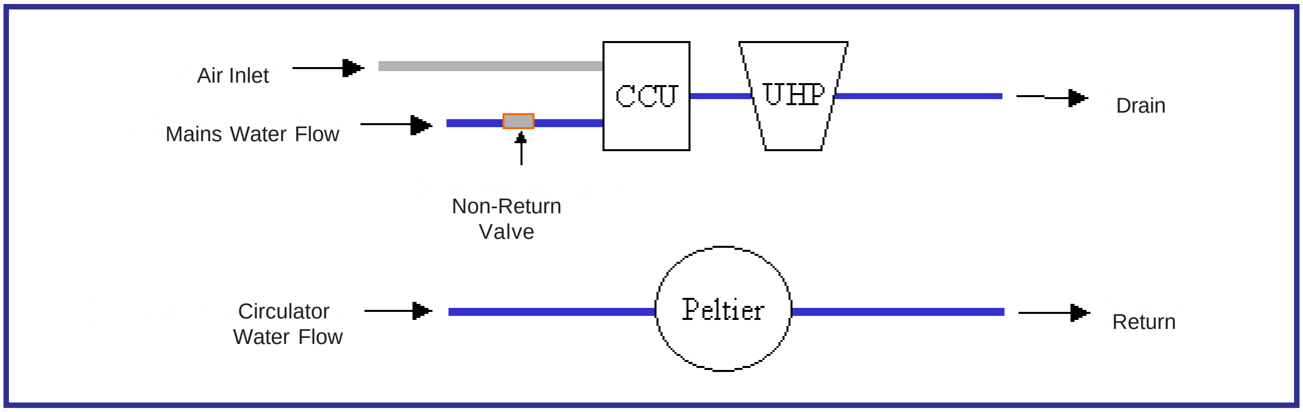


Figure 7.10
Cooling Water Configuration 3
Fluid Circulator Supplying Peltier, Mains Water Supplying Upper Heated Plate

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 7.11 to the right below).

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 7.11
The Cooling Control Unit

Figure 7.12
Non-Return Valve
(For use with mains water supply only.
Note the direction of flow through the valve.)

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.

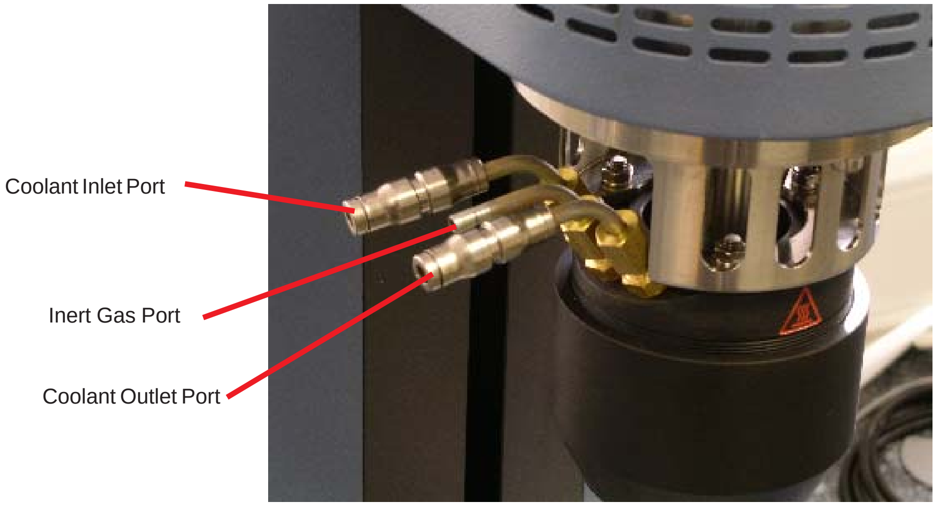


Figure 7.13
Coolant and Inert Gas Connections for the UHP

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 7.2 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 7.2

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 froid 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 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.

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

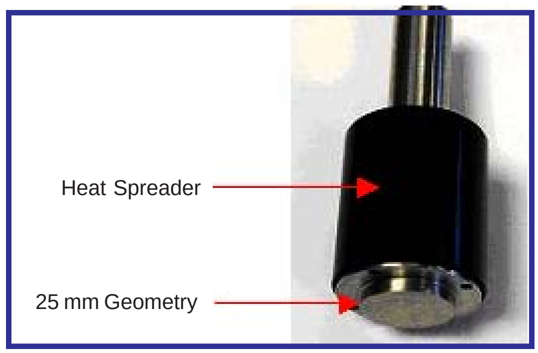


Figure 7.14
Upper Geometry Holder
(Shows the cylindrical heat spreader with a 25 mm diameter geometry in place.)

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 the figure to the right will appear.

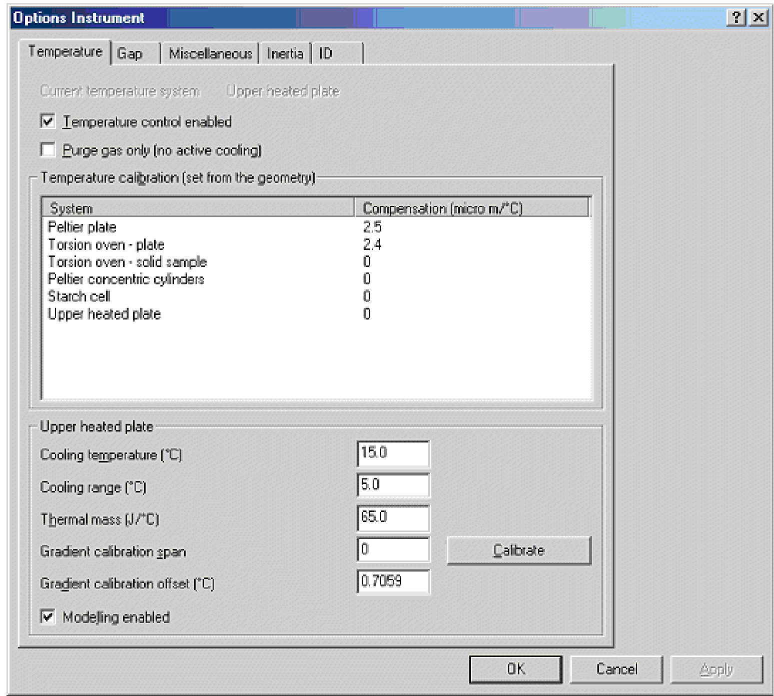


Figure 7.15
UHP Configuration Window

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. For the vortex air cooler, use the value given in Table 7.3

- **Cooling range:** This is inversely proportional to the flow rate. For the vortex air cooler, use the value in Table 7.3. Typical values are given in the table below.

Table 7.3

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
Vortex Air Cooler	15 °C	--	100 °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.

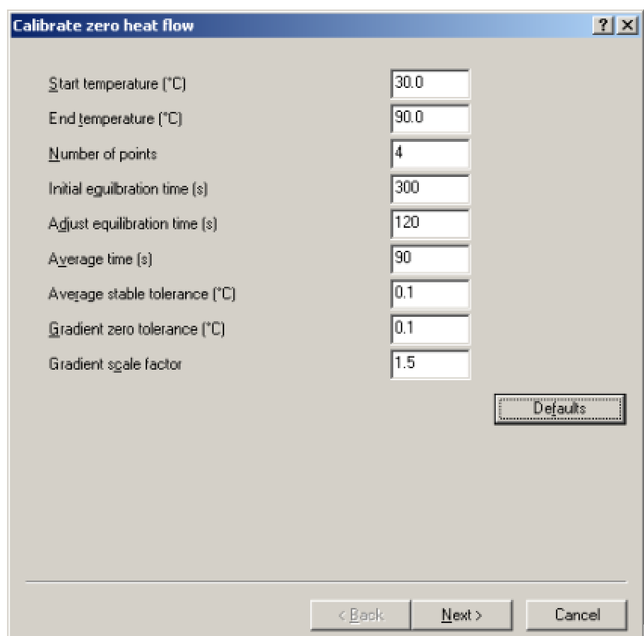


Figure 7.16
UHP Calibrate Zero Heat Flow Window

1. Under the **Options** menu click **Instrument** and then the **Temperature** tab. The window, shown in Figure 7.15 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 7.16 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.

- **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 7.18 below.

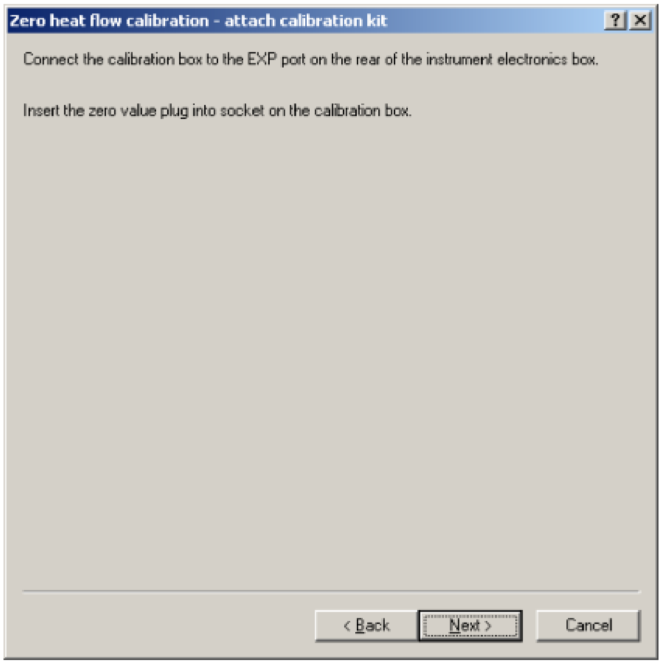


Figure 7.17
Zero Value Determination Instructions

Figure 7.18
Upper Heated Plate Calibration Box (Left) and Zero Value Plug (Right)

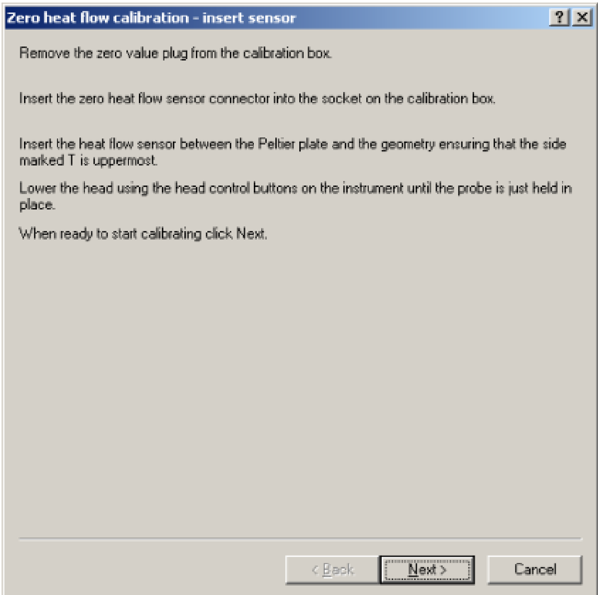


Figure 7.19
UHP Calibration Instructions

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 7.20 below.

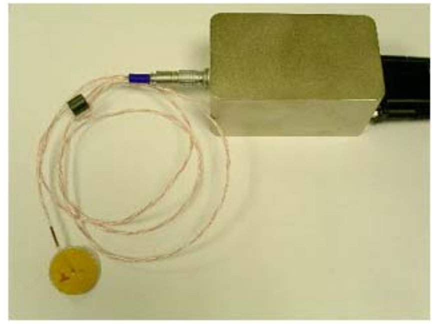


Figure 7.20
Zero Heat Flow Sensor Connected to Calibration Box (Note the **T** on the upperside of the sensor.)

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 7.21.

Figure 7.21
Positioning the Heat Flow Sensor

7. Click **Next** to begin calibrating as instructed (see Figure 7.19, above left). When the calibration is complete the results will be displayed as shown in Figure 7.22 (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.

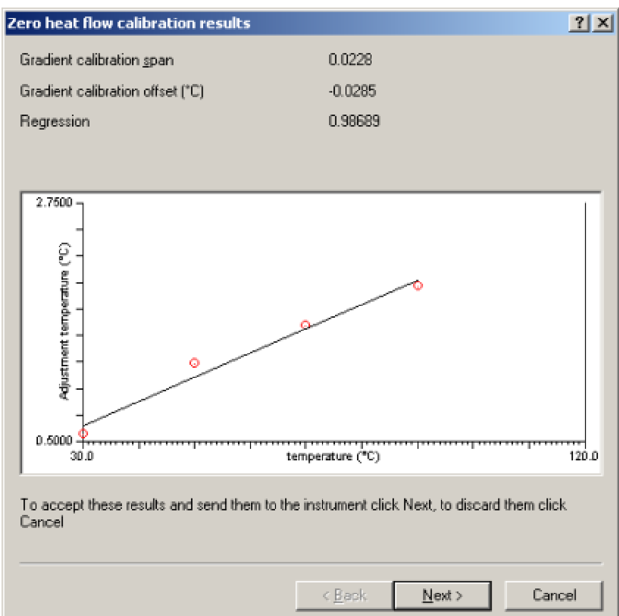


Figure 7.22
Results of UHP Calibration

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 7.23).

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.

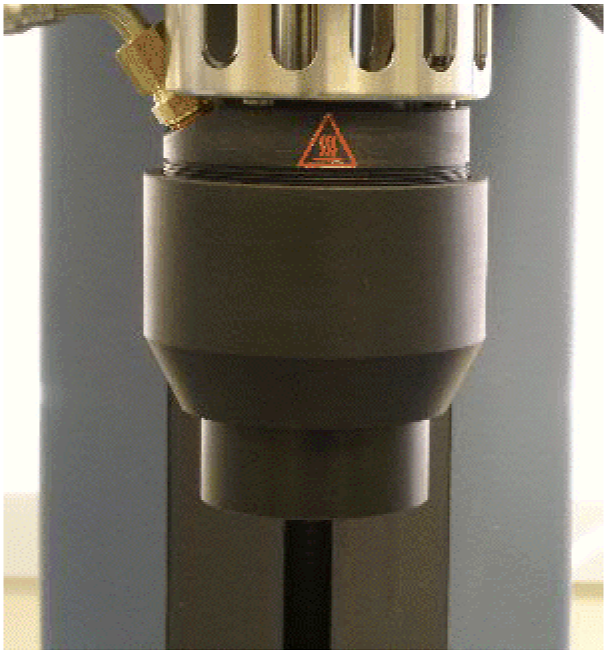


Figure 7.23
Air-Bearing Clamp

Using an Inert Gas Atmosphere

Many samples experience oxidation at elevated temperatures—an atmoshere 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.

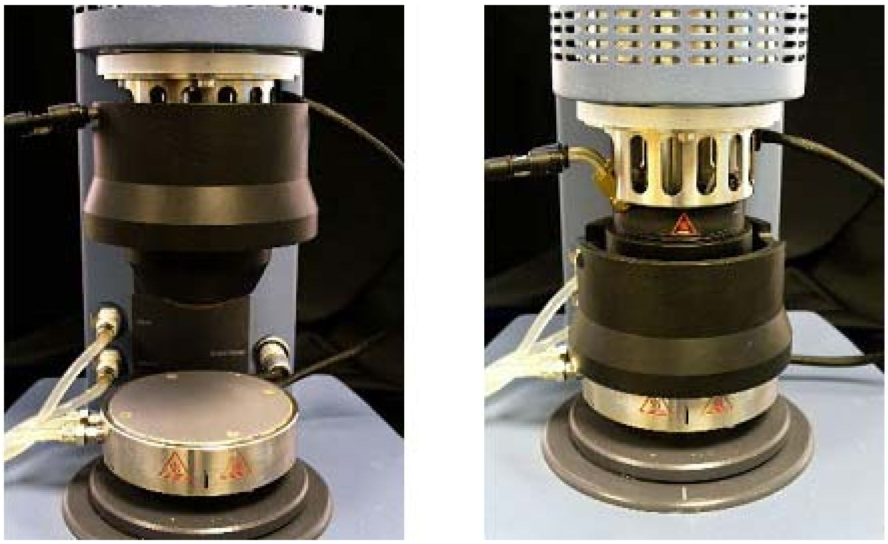


Figure 7.24
Sample Cover
Up Position (Left) and Down Position (Right)

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

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

Chapter 8

The Pressure Cell

Overview

WARNING: TA Instruments' Pressure Cell is designed for use at temperatures up to 150°C and pressures up to 138 bar (2000 psi). At all times during the use of the cell, wear safety glasses and clothing that afford adequate protection against the sample under test, and the temperature and pressure used. At other than ambient temperature, the outer surfaces of the cell may become very hot or cold. When operating at these temperatures, wear gloves that afford adequate protection against the surface temperature of the pressure cell and its fittings.

The Pressure Cell is used with the standard concentric cylinder, Peltier-controlled, heating jacket. A copper sheath is fitted to the cell to ensure good heat transmission between the jacket and the cell.

The Pressure Cell may be used either in *self-pressuring mode*, in which the pressure is produced by the volatility of the sample, or in *external pressurization mode*, with an applied pressure of up to 138 bar (2000 psi). In this chapter, the pressure cell assembly and operation for both modes are described.

NOTE: For external pressurization, the user of the cell is required to provide a high-pressure source, and suitable pressure-rated connections to a 1/8-inch or 1/4-inch NPT female fitting.

Specifications

Operating Specifications

The specifications for the standard pressure cell concentric cylinders are:

Stator inner radius:	14.00 mm
Rotor outer radius:	13.00 mm
Cylinder immersed height:	44.00 mm
Gap:	3500 μm (recommended)
Backoff distance:	3500 μm
Geometry inertia:	92.00 μN.m.s2 (approximate)
Sample volume:	9.5 ± 0.5 ml
Temperature range:	-10 to 150°C
Maximum applied pressure:	138 bar (2000 psi)
Maximum pressure (self-pressurizing):	5 bar (72.5 psi)
Torque range	about 100 μN.m to 0.2 N.m
Maximum angular velocity:	50 rad/s
Seal construction:	DuPont Kalrez®

Safety Specifications

Over pressure rupture disk:	172 bar (2500 psi)
Hydraulically tested to:	414 bar (6000 psi)

Operational Limits

CAUTION: To prevent sample entering the upper part of the cell and contaminating the bearings, the cell should not be used above the limits given below. Exceeding these limits may also cause mechanical damage to the cell.

- Maximum Angular Velocity: 50 rad/s
- Maximum Sample Viscosity: The geometry should not be forced into the sample. Light hand pressure should be all that is required.
- Maximum Frequency: 50 Hz (314 rad/s)

NOTE: The quality of data obtained using the pressure cell cannot be expected to match that obtained when conventional measuring systems are used with the rheometer. Some of the normal calibration routines are not relevant to, or cannot be used with the Pressure Cell. Alternative calibration routines are described in this chapter.

Pressure Cell Components

The Pressure Cell consists of four main component assemblies. These components include the pressure cell cup, the concentric cylinder rotor, the magnet assembly, and the pressure manifold. Figure 8.1 shows a schematic cross section of the pressure cell cup, rotor, and magnet assemblies, and Figure 8.2 shows a fully configured Pressure Cell installed on an AR Rheometer. The following section will discuss these four components individually.

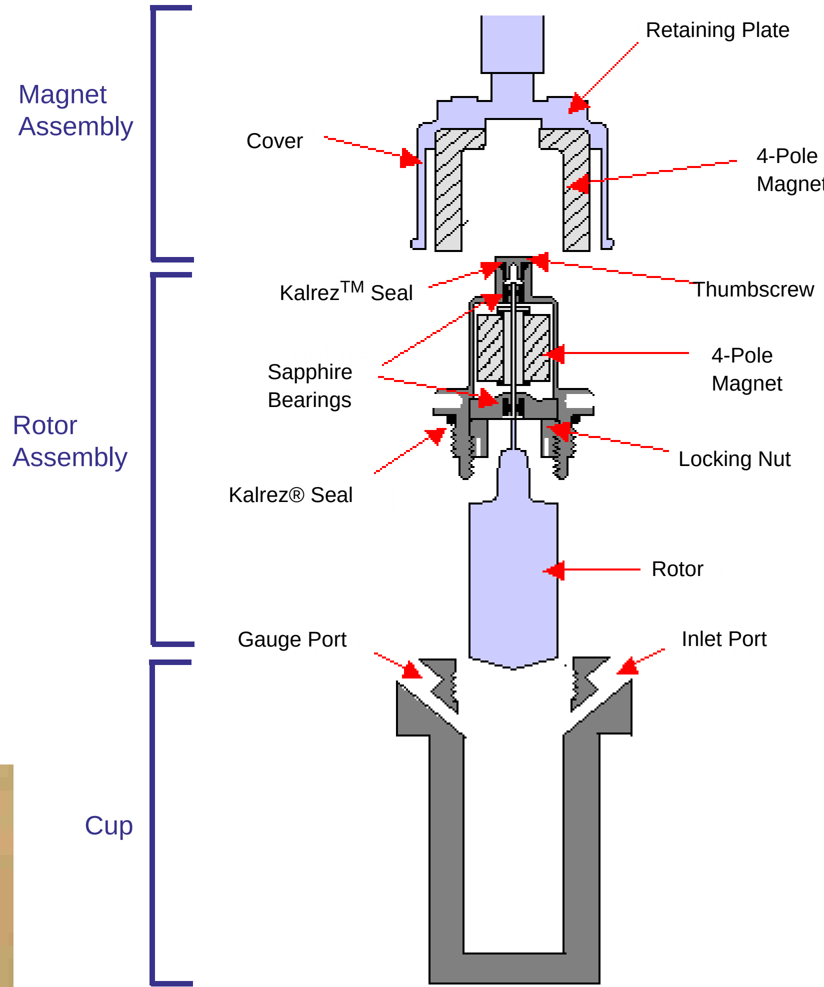


Figure 8.1
Cross Section Schematic

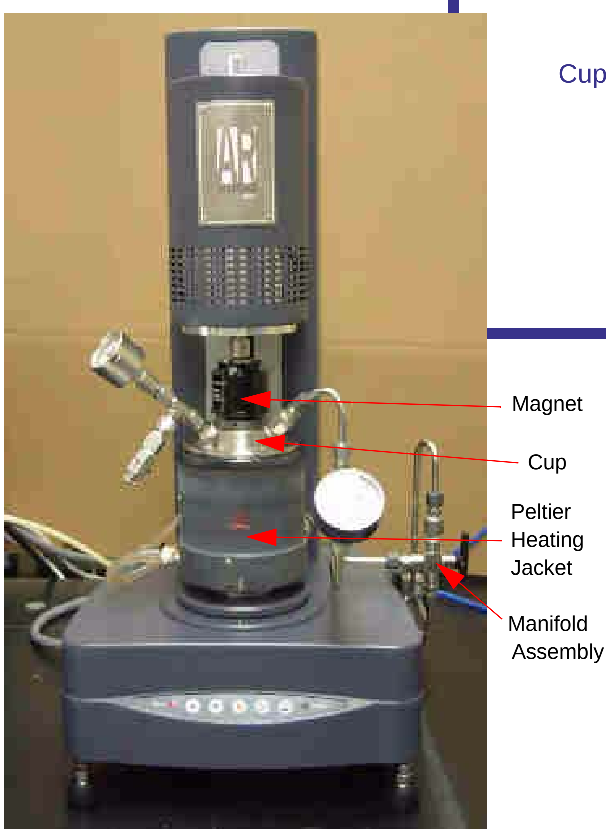


Figure 8.2
Pressure Cell on AR Rheometer

The Pressure Cell is shown assembled on the AR rheometer, with the instrument head in the DOWN position, in Figure 8.2 to the left.

The Pressure Cell Cup

The Pressure Cell Cup contains the sample fluid. It is inserted into the Peltier jacket, which mounts on the rheometer using the Smart Swap™ connection. A copper sheath ensures good heat transmission between the jacket and the cup. There are three ports on the cup, which are identified by engraved labels.

NOTE: When installing NPT fittings use Teflon® thread sealing tape.

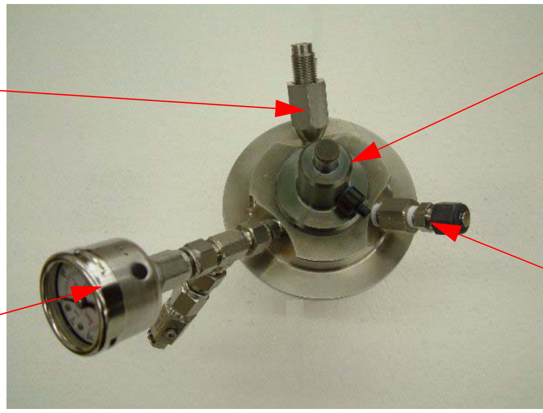


Figure 8.3
Pressure Cell Cup with Rotor Assembly

CAUTION: Do not attempt to attach or detach any fittings to or from the cell while it is mounted on the rheometer. Doing so can cause damage to the instrument.

The Inlet Port

The Inlet Port, which is used in the external pressurization mode, is where the compressed gas is introduced to the cup. A pressure manifold is supplied that attaches to the inlet port using a compression connector. A pressure snubber is fitted between the port and the high-pressure line to slow the pressure build and prevent sample from entering the line.

The Pressure Gauge Port

This port is fitted with a pressure gauge which indicates the pressure within the cell, and a relief valve. This valve is only intended to be used when the pressure from the cell cannot be relieved in the usual way (see "Pressurizing and Depressurizing the Cell," found later in this chapter). You will need a 5/8-inch open or box-end wrench to hold the valve body and a 7/16-inch wrench to open the valve.

Figure 8.4
Pressure Gauge Port
(Gauge and Valve Shown)

Safety Relief Port

The Safety Relief Port is equipped with a rupture disk assembly that is designed to relieve excessive cup pressure. At excessive internal pressure, the rupture disk breaks and propels the internal atmosphere out of the cup.

WARNING: Do not operate the pressure cell without the safety relief fitting in place. Do not remove the rupture disc from the safety valve fitting, as this may cause the pressure cell to crack during an overpressure condition, resulting in damage and personal injury. The rupture disc should only be replaced by a qualified TA Instruments Service Representative.

CAUTION: You MUST install the Safety Relief Port with the Rupture Disk such that it is pointed to rear of the AR-G2 and away from the operator. This will prevent sample material from being ejected toward the operator in the event of an over-pressure situation.

Rotor Assembly

The rotor assembly contains the Concentric Cylinder rotor, which is mounted on a shaft that is radially supported by two sapphire bearings located under the rotor assembly cap. Also attached to the shaft is a four-pole magnet. The rotor assembly installs into the cup using a threaded mount, and seals with a Kalrez® seal. A second Kalrez seal is seated between the cap and the thumbscrew.

Figure 8.5
Pressure Cell Rotor Assembly with Cap Off (Left) and On (Right)

CAUTION: Prior to use, ensure that the two Kalrez seals are installed and are in good condition. Replace, if damaged, with seals provided by TA Instruments only.

Magnet Assembly

The magnet assembly attaches to the rheometer's rotating spindle, and then lowers over the rotor assembly. The spindle collar of the magnet assembly includes an insert adapter. The adapter insert should remain in the collar for use with AR2000 or AR2000ex rheometers. If using the pressure cell with an AR-G2 rheometer, take the adapter insert out of the spindle collar by removing the two Phillips (or cross head) screws extending for the outer surface of the spindle collar. See the figure below. Like the rotor assembly, the magnet assembly contains a 4-pole magnet. When the spindle and magnet assembly are rotated, the attraction between the two 4-pole magnets produces a corresponding rotation of the rotor. There is no physical contact between the two assemblies.

Figure 8.6
Magnet Assembly with Cover

CAUTION: Do not place magnetic storage media near the magnet assembly, as it contains a powerful magnet capable of destroying magnetically recorded material.

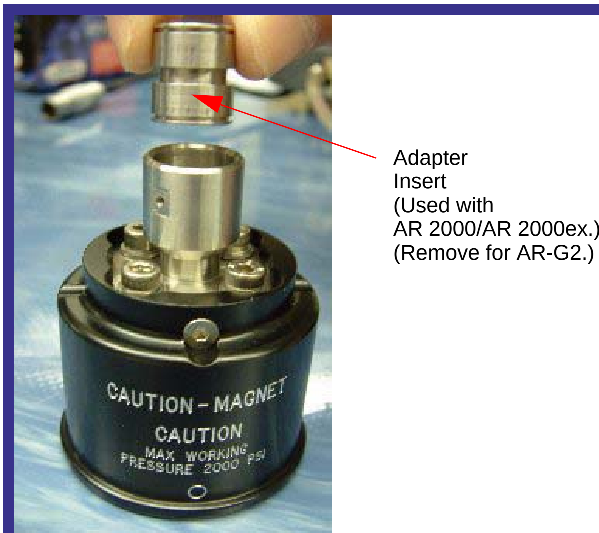


Figure 8.7
Magnet Assembly with AR 2000/ AR 2000ex Adapter Insert

Pressure Manifold

The Pressure Cell includes a high-pressure manifold assembly that is connected to the rheometer frame. The rigid piping pressure manifold provides strain relief between the pressure cell and external high-pressure connections. It also includes necessary valves and gauges for safely pressurizing and depressurizing the cell. It is a critical part of the pressure cell assembly and the pressure cell should not be operated without the manifold in place. The pressure manifold, shown in Figure 8.8 to the right, includes the following:

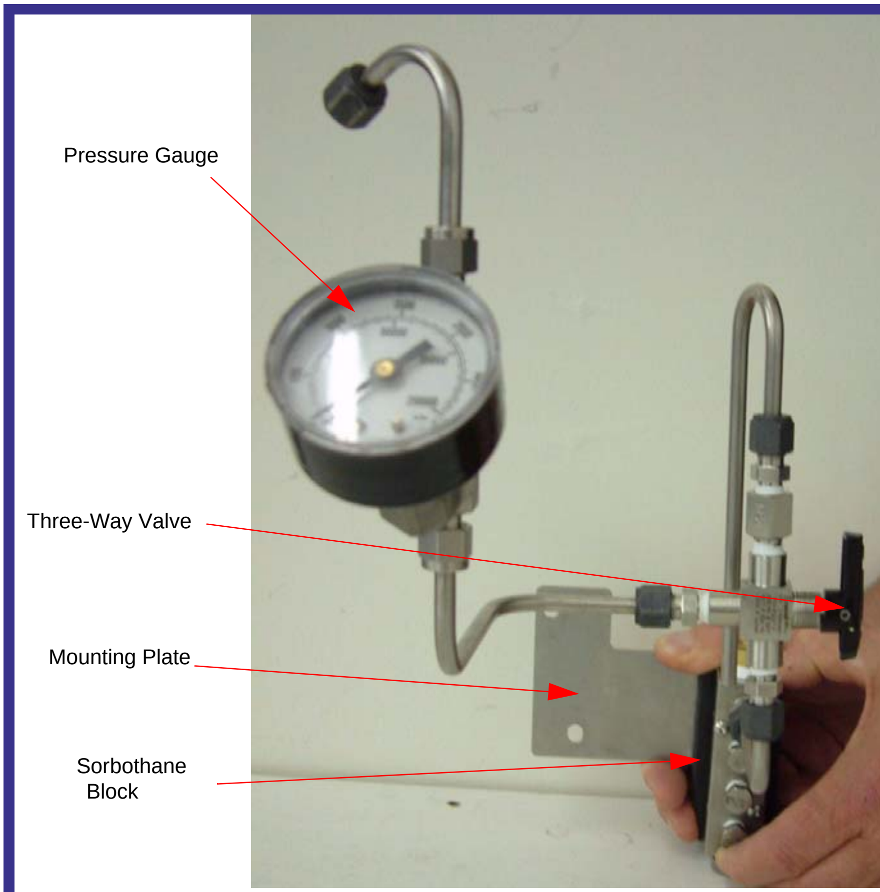


Figure 8.8
Pressure Manifold Assembly

- Mounting plate and Sorbothane block. The Sorbathane block is a flexible material that provides flex between the rigid pressure cell piping assembly and the rheometer frame.

- 1/8-inch and 1/4- inch female NPT fittings for high-pressure connections.

- Three way valve for pressurizing, maintaining cell pressure, and depressurizing the cell.

- Pressure gauge for monitoring pressure in the cell.

Requirements for External Pressure Source

Fittings are provided for external pressurization up to 138 bar (2000 psi). A high-pressure source must be supplied with 1/8-inch or 1/4-inch NPT male fittings for connection to the manifold supplied by TA Instruments. In addition, a means of isolating the source from the manifold, and of relieving the pressure in the line from the source to the manifold should be provided. Figure 8.9 below shows a typical set up for external pressurization.

WARNING: Only use TA Instruments' high-pressure manifold when operating the pressure cell. Ensure that the manifold can be isolated from the high-pressure source provided by the user, and that there is a pressure vent valve in the line between the source and the manifold.

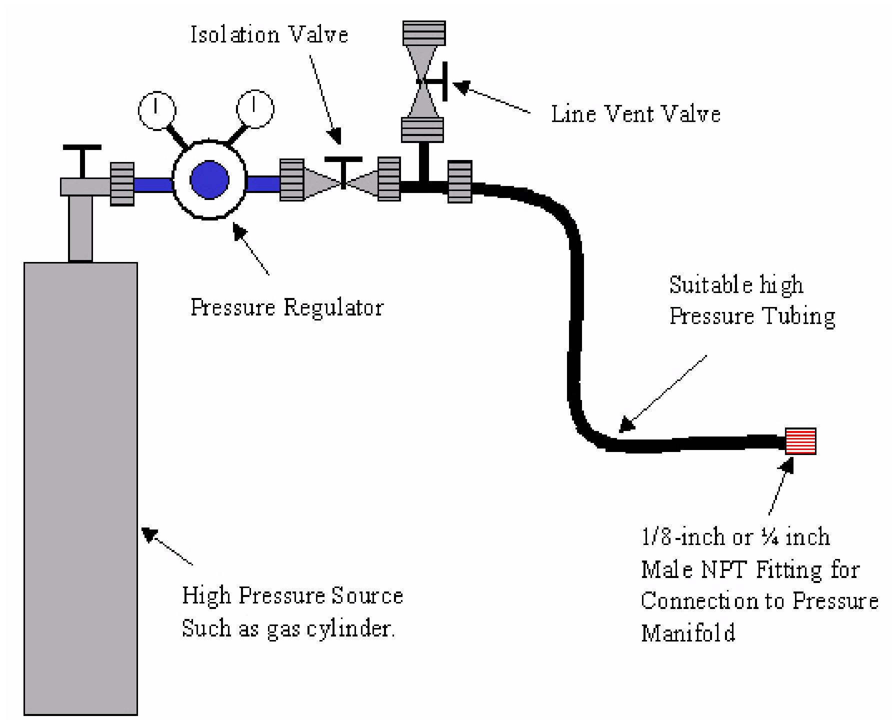


Figure 8.9
Typical Setup for External Pressurization

Installing and Using the Pressure Cell

The AR-G2 and AR2000/ex Pressure Cell is shipped with the Rotor installed in the pressure cell cup, and the high-pressure piping manifold fully assembled. Installing the AR Pressure Cell from it's initial shipped configuration requires some disassembly. Disassembly instructions are as follows:

- **Step 1**: Unpack the preassembled pressure cell cup and rotor. Using the spanner (Tommy bar) provided, remove the rotor assembly from the pressure cell cup, and carefully place the rotor in a safe location. Remove foam packing material from Cup.

> NOTE: All the compression fittings that are used on the manifold assembly have a specific tightening procedure for the first time the nut is tightened on the tubing during the manufacturing process. Subsequent disassembly and remake of any fitting should be done by first putting a **reference mark** on the nut and the body of the fitting before loosening the fitting. After reassembly of the fitting to a finger tight condition, the nut should then be tightened with a wrench so that the mark on the nut aligns with the mark on the body of the fitting. A second wrench should be used to hold the body of the fitting in place while turning the nut.

- **Step 2**: Unpack the preassembled piping manifold assembly. Using a wrench, remove the furthest extending angled pipe from the Tee fitting joining the gas inlet and pressure gauge.

Figure 8.10 below shows the disassembled configuration.

The steps on the next several page provide the instructions needed to install and use the pressure cell.

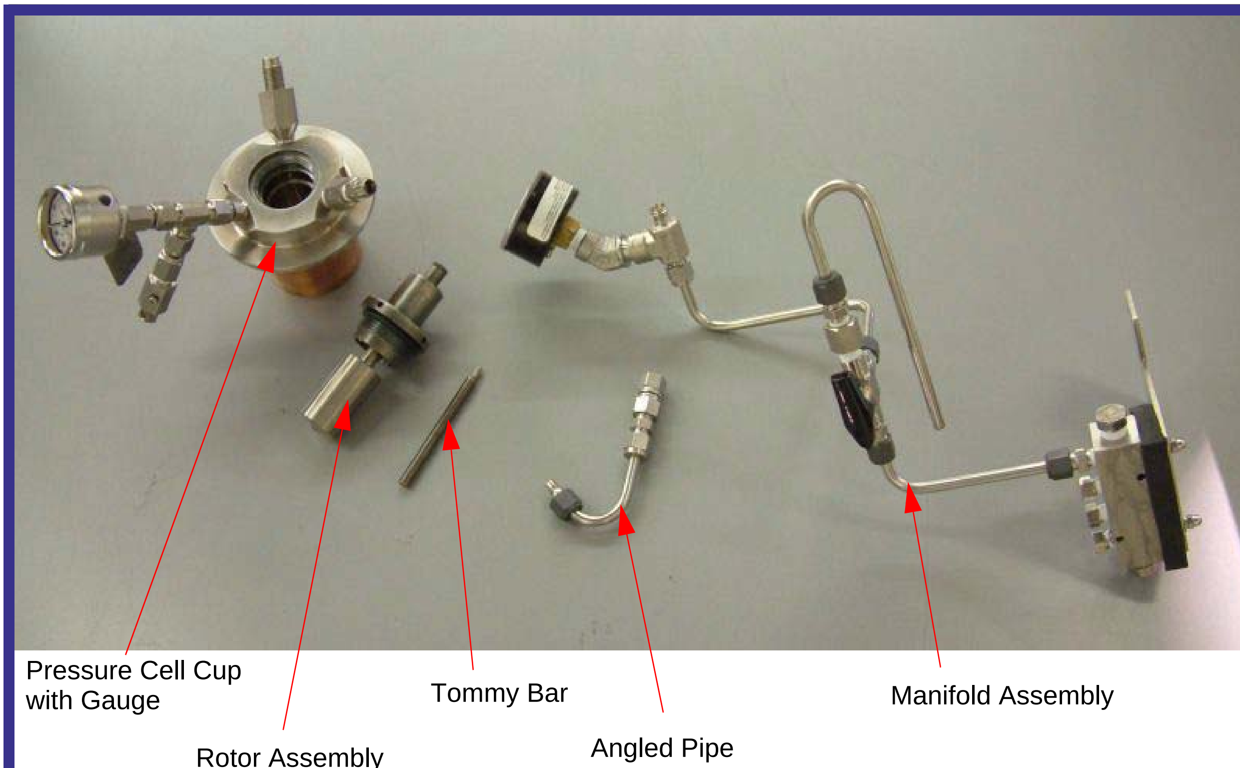


Figure 8.10
Entire Pressure Cell Configuration Disassembled

Step 1: Install High-Pressure Piping Manifold

The pressure manifold attaches to the lower right rear as viewed from front of rheometer or left lower corner when facing rear of the AR rheometer, as shown in Figure 8.11.

Attach the mounting plate and Sorbothane block to the two M5 using the cap head screws provided, as shown in Figure 8.12 below.

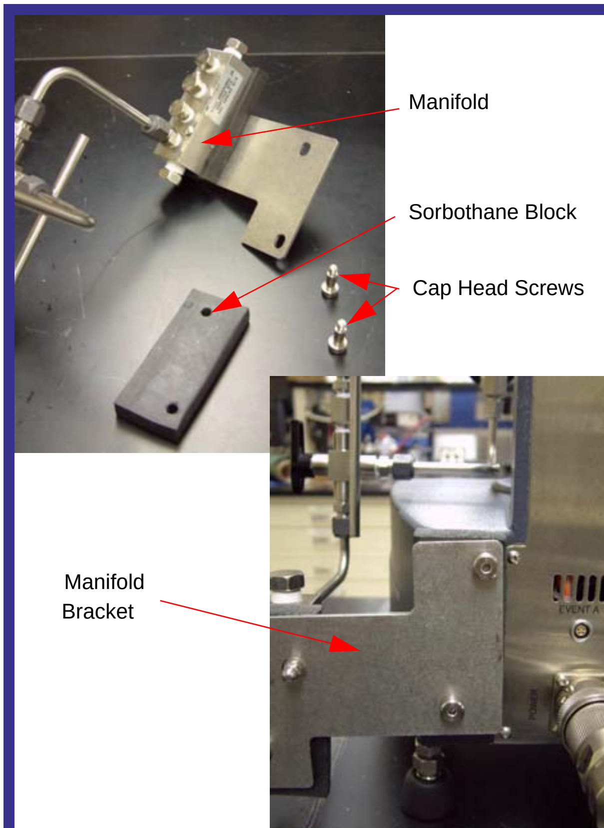


Figure 8.12
Manifold Installed

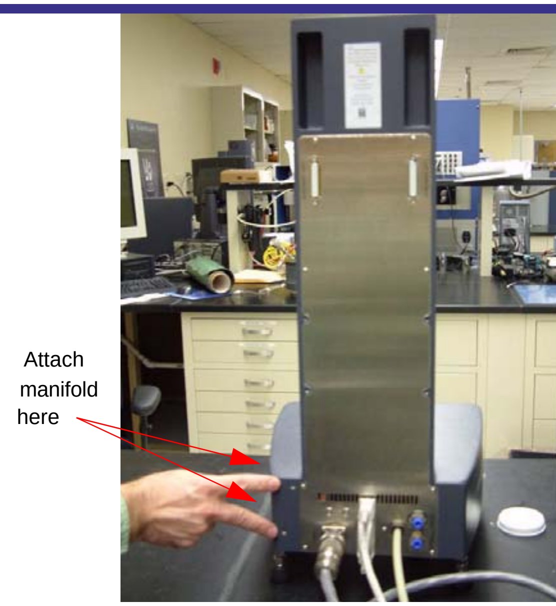


Figure 8.11
Attach Manifold to Back of Rheometer

Step 2: Install and Configure Pressure Cell Cup and Rotor

Follow the instructions below to both install and configure the Pressure Cell Cup and Rotor:

1. Locate the Peltier Concentric Cylinder Jacket. Remove the Peltier Jacket, if it is installed on the AR Instrument.

2. Remove any Peltier Concentric Cylinder cups and remove the two knurled screws that fasten the standard Peltier cups in place (Note the standard knurled screws can not be used with the Pressure Cell).

3. Insert the Pressure Cell cup into the jacket, with the Safety Relief valve facing to the rear of the cup. (Note the "Hot" symbol and white Smart Swap alignment line are markings on the front of the Peltier Jacket). Fix the cup in position in the jacket using the two hex head screws and hex keys provided with the Pressure Cell. See Figure 8.13 to the right.

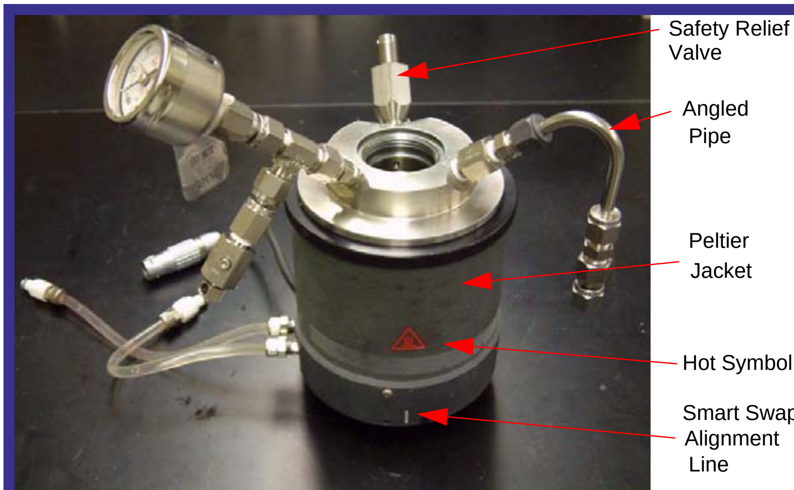


Figure 8.13
Pressure Cell Cup Installed in Peltier Jacket

4. Connect angled high-pressure pipe to the cell inlet port, ensuring that the straight part of the pipe is vertical as shown in Figure 8.13. Tighten the compression connector as directed in the NOTE on page 93.

5. Locate the rotor assembly. Place the small magnet onto the rotor assembly, as shown in Figure 8.14, such that the small magnet is vertically aligned with the scribe line etched on the collar on the rotor assembly.

6. Hand-tighten the rotor assembly into the cup. Fully tighten the rotor until flush with the cup using the wrench (also called a Tommy bar).

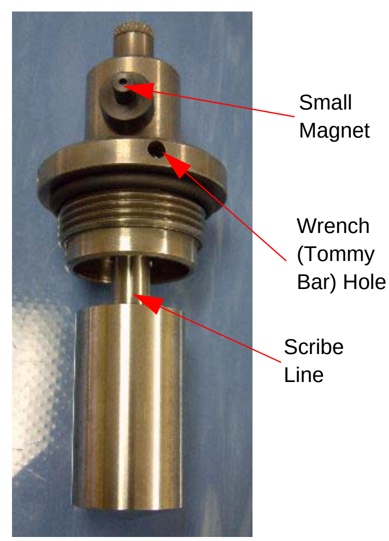


Figure 8.14
Rotor Assembly
with Small Magnet

Figure 8.15
Orientation of Small Magnet

7. Position the small magnet on the rotor to face in the front center of the Peltier Jacket as shown in Figure 8.15 to the left.

8. Install Peltier Jacket on rheometer using Smart Swap Connectors.

9. Install the Magnet Assembly onto the shaft of the rheometer.

10. Rotate the draw rod so the magnet assembly reference mark is aligned with the small magnet on the Rotor. Ensure that the reference mark on the upper geometry remains aligned with the small magnet by lightly holding the rheometer draw rod and begin lowering the rheometer head as shown in Figure 8.16

11. At a gap of about 20 mm between the shoulder on the rotor assembly, and the underside of the upper magnet assembly, the magnets in the upper assembly will engage with those in the rotor assembly as shown in Figure 8.17. (A small noise will be heard when this happens and a change of a few Newtons will be seen in the normal force reading.). Immediately stop moving the rotor assembly down and remove the small magnet.

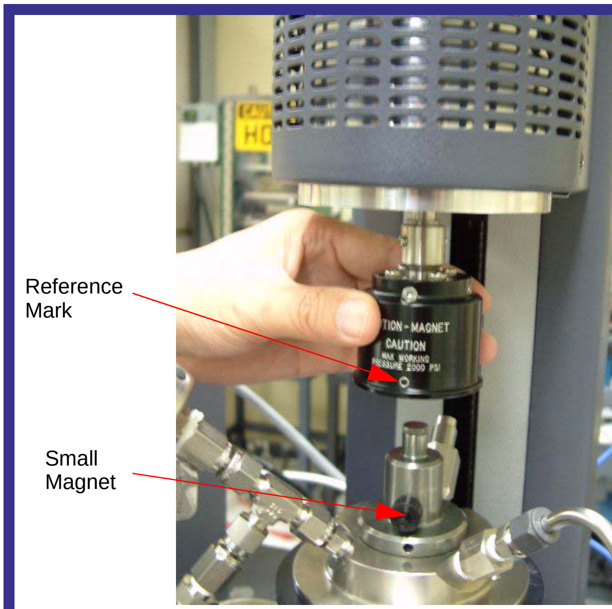


Figure 8.16
Aligning the Reference Mark

Figure 8.17
Magnets Engaging

Step 3: Positioning Gap and Pressure Cell Calibrations

Once the Pressure Cell cup and rotor have been installed, you will need to position the gap and perform the calibrations as directed in this section.

CAUTION: The standard calibration routines used by Rheology Advantage for zero gap, geometry inertia and bearing friction are not suitable for use with the pressure cell. When the pressure cell is selected as the measuring geometry, these routines are either disabled or are replaced by more appropriate routines. Do not attempt to use or calibrate the pressure cell unless this geometry is selected.

1. Ensuring that the pressure cell geometry is selected in the software, find the gap zero position. Do not request the instrument to raise the head to the backoff distance. Set a gap of 3500 μm.

2. When the Pressure Cell is the selected geometry on an AR2000 or AR2000ex, the Gap Zero Mode of normal force with a value of 5N will be used. This will override any other settings in Rheology Advantage software.

3. Conduct the Bearing Friction Calibration. The bearing friction routine used when the Pressure Cell is selected as the geometry is slightly different from the standard routine. The calibration should be conducted at a geometry gap of 3500 μm. The bearing friction calibration must be done again when another measuring system is used. A typical value for the Pressure Cell should be between 8 and 15 μN.m / (rad/s). This is about ten times higher than for other geometries.

4. Map the Air Bearing. Perform a rotational mapping at a gap of 3500 μm using the Standard mapping routine.

   NOTE: DO NOT USE PRECISION OR EXTENDED MAPPING ROUTINES WITH THE PRESSURE CELL.

   CAUTION: It is important that the bearing is re-mapped before any other measuring system is used.

   NOTE: When changing to other geometries, Rheology Advantage does not restore the previous settings. However, the mapping table is cleared and the bearing friction is reset to zero. Any functions that were previously unavailable are reactivated and the gap zero mode settings are restored, because the settings were not overwritten.

5. Check the Cell by running peak-hold test at 0.05 rad/s and a duration time of 126 sec. The peak-to-peak residual torque should not be larger than 100 μN.m.

Step 4: Loading a Sample

Samples are loaded in the pressure cell after the cell is set up and calibrated. The following steps will detail the sample loading procedure.

1. Rotate the drawrod so the reference mark on the magnet assembly is facing the front of the instrument. Raise the rheometer head high enough to place the small magnet on the rotor. Once the small magnet is in place, raise the rheometer head to the maximum height.

   NOTE: DO NOT REMOVE MAGNET ASSEMBLY FROM THE RHEOMETER HEAD. IF IT IS REMOVED, THE MAPPING WILL NO LONGER BE AS EFFECTIVE, CAUSING AN INCREASE IN RESIDUAL TORQUE.

2. Remove the Peltier jacket from rheometer.

3. Leaving the small magnet in place, gently remove the rotor from the cup.

4. Load the sample into the cup. For very viscous samples, you may find it easiest to weigh the sample in the cup, if the sample density is known (this can be done after removing the cup from the jacket).

   NOTE: Volume is 9.5 ± 0.5 mL.

5. Ensure that the small magnet is still aligned with the mark on the rotor assembly.

6. Replace the rotor assembly and fully tighten.

7. Replace the Peltier jacket onto the Smart Swap Base of the rheometer.

8. Rotate the draw rod so the magnet assembly reference mark is aligned with the small magnet on the Rotor. Ensure that the reference mark on the upper geometry remains aligned with the small magnet by lightly holding the rheometer draw rod and begin lowering the rheometer head as shown in Figure 8.18.

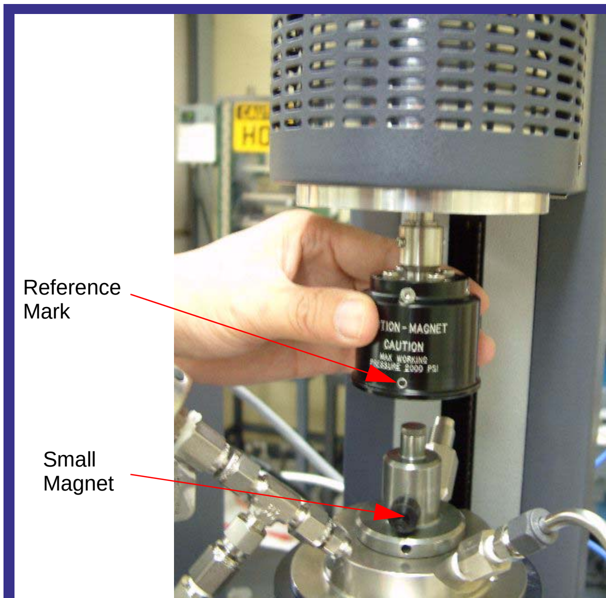


Figure 8.18
Aligning the Reference Mark

Figure 8.19
Magnets Engaging

9. At a gap of about 20 mm between the shoulder on the rotor assembly and the underside of the upper magnet assembly the magnets in the upper assembly will engage with those in the rotor assembly as shown in Figure 8.19. (A small noise will be heard when this happens and a change of a few Newtons will be seen in the normal force reading.). Immediately stop moving the rotor assembly down and remove the small magnet.

10. Lower the instrument head to the geometry gap (default 3500 µm). Do not zero the gap.

Step 5: Align Manifold and Make Manifold Connections

After the sample is loaded, you will need to align the manifold as follows. The pressure manifold should only be connected and disconnected from the cup assembly at the bottom of the angled pipe connected to the cup as shown in Figure 8.20. Please refer to the NOTE on page 93.

1. Prior to connecting the pressure manifold to the angled pipe, slacken off to finger tight the compression connectors on the manifold in order to easily align the manifold with the pipe mounted on the cup. DO NOT USE EXCESSIVE FORCE TO POSITION THE PRESSURE MANIFOLD.

2. Finger-tighten ONLY the manifold to the angled pipe mounted on the cup, using the connector indicated in Figure 8.21.

Figure 8.20
Place to Connect/Disconnect Manifold

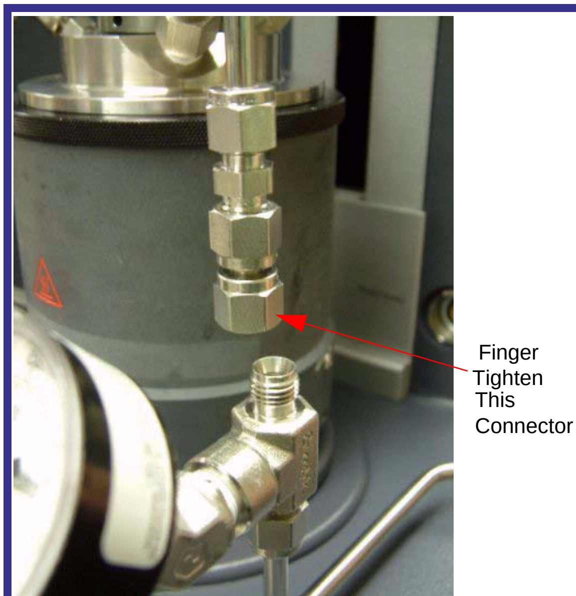


Figure 8.21
Connector Used to
Connect/Disconnect Manifold

3. Once this fitting is finger tight, fully tighten all other compression fittings as directed in the NOTE on page 93.

4. Finally, fully tighten the angled pipe to the manifold as directed in the NOTE on page 93.

CAUTION: To avoid putting excessive force on the pressure cell, make sure that connection or disconnection between the pressure cell and the manifold is made at the breakage point compression connector only (see Figures 8.20 and 8.21. Connection or disconnection should be made at no other point while the pressure cell is mounted on the rheometer.

NOTE: To disconnect the pressure cell from the manifold, ensure that both the cell and the manifold are depressurized. Then disconnect the compression connector indicated in Figures 8.20 and 8.21.

WARNING: Before disconnecting the pressure cell from the manifold, ensure that neither the cell nor the manifold is pressurized, and that both are cool enough to touch.

Step 6: Pressurizing/Depressurizing the Cell and Running Experiments

When the cell is fully installed, it can be operated in either *self-pressurization mode*, in which the pressure is due to the volatility of the sample, or in *external pressurization mode*, in which the pressure is provided by an external source. The pressure achieved when the cell is used in self-pressurizing mode will depend on the sample and temperature. As a guide, the vapor pressure of water at 150°C is about 4.76 bar (69 psi.).

When the cell has been assembled correctly as directed in this chapter, it can be pressurized. The manifold contains a three-way valve. The three positions for the valve are shown in Figure 8.22.

Vertical Up—Pressure Relief

Connection between the pressure cell and atmosphere.

Vertical Down—Pressure Build (external pressurization mode)

Connection between the pressure cell and the connector block on the manifold.

Horizontal—Off

Isolation between the pressure cell and the connector block on the manifold.

Figure 8.22
Three-Way Valve Positions

Running Experiments in Self-Pressurization Mode

After the manifold is installed, all fittings have been tightened, and the sample has been loaded, follow these steps to run an experiment in the self-pressurization mode.

1. Make sure the three-way valve on the manifold is set to the OFF position (horizontal).

   NOTE: The secondary pressure relief valve on the Pressure Gauge Port should only be opened if the primary pressure relief passage becomes clogged. If this valve was opened, make sure it is closed.

2. Set the testing temperature from the software.
3. Program the test procedure.
4. Run the experiment.
5. After running the experiment, depressurize the cell by following these steps:
   a. Ensure the the pressure cell is isolated from the high pressure source.
   b. When the experiment is complete, the temperature should be set back to 25°C (ambient temperature) before attempting to relieve the pressure in the cell.
   c. Once the cell has cooled, set the three-way valve on the manifold to the "Pressure Relief" position (arrow pointing upward). Note that the pressure may reduce very slowly, due to the snubbers in the manifold.

   NOTE: There are two snubbers in the manifold, one of which is located on the cell itself and the other in the vent line, as described earlier in this chapter. These are used to slow the build and relief of pressure in or from the cell. The snubbers may become clogged with sample, preventing the cell pressure from being properly relieved (this problem is more likely to occur when external pressurization is being used). You must maintain the cleanliness of the snubbers, ensuring that gas can pass through them. You can verify that they are operating properly by pressurizing and depressurizing the cell without a sample. If there is any doubt concerning the cleanliness or operation of the snubbers, they should be replaced.

   NOTE: The pressure relief valve on the cell's Pressure Gauge Port can also be used to relieve the pressure in the cell. This should only be done if the three-way valve on the manifold cannot be used, if, for example, the snubbers have become blocked. The cell should also be at room temperature before using this valve, and appropriate gloves and safety glasses should be worn.

The pressure gauge located on the Pressure Gauge Port and the pressure gauge on the manifold can be used to help verify that the snubbers are not clogged. After relieving the pressure in the cell using the manifold and three-way valve, the two gauges should read zero (0) pressure. If the snubbers are clogged, the pressure gauge in the Pressure Gauge Port may still register pressure. If that condition occurs, use the relief valve on the Pressure Gauge Port to release pressure from the cell.

CAUTION: Although the pressure gauge on the cell may to appear to register zero, the level of pressure in the cell may still be above ambient.

- Disconnect the pressure cell from the manifold at the designated disconnection point at bottom of the angled pipe.

- Raise the head and decouple the magnet assembly and the rotor, and remove the Peltier jacket from the rheometer using the procedures outlined previously in "Step 4: Loading a Sample."

- Remove the cup and rotor for cleaning. See "Maintaining the Cell" for details.

Running Experiments in External Pressurization Mode

NOTE: If the maximum pressure of the external pressure source is greater than 138 bar (2,000 psi), (*e.g*. N2 tank), the source must be regulated to a maximum value of 138 bar (2000 psi).

After the manifold is installed, all fittings have been tightened, and the sample has been loaded, follow these steps to run an experiment in the external pressurization mode.

1. Make sure the three-way valve on the manifold is set to the OFF position (horizontal).

   NOTE: The secondary pressure relief valve on the Pressure Gauge Port should only be opened if the primary pressure relief passage becomes clogged. If this valve was opened, make sure it is closed.

   WARNING: Before applying high pressure, check for leaks at low pressure. Raise the pressure of the cell, gradually, making frequent leak checks.

2. Regulate the external pressure source to the pressure required for the experiment. THIS PRESSURE CANNOT EXCEED 138 bar (2,000 psi).

3. Gently pressurize the cell by slowly opening the three-way valve on the manifold to the pressure build position (arrow pointing vertically downwards). Check the pressure cell to make sure there are no leaks. If it is free from leaks at low pressure, raise the pressure gradually, making frequent leak checks using a liquid leak test material.

4. During operation, the pressure cell should be isolated from the high-pressure source. Set the three-way valve on the manifold to OFF (arrow horizontal), and relieve the pressure in the line between the high-pressure source and the connector block. If the cell is free from leaks, the pressure in the cell will be maintained.

   WARNING: During operation, isolate the pressure cell from the high-pressure source, and relieve the pressure in the line between the source and the manifold.

5. Set the testing temperature from the software.

6. Program the test procedure.

7. Run the experiment.

8. After running the experiment, depressurize the cell by following these steps:

   a. Ensure the the pressure cell is isolated from the high pressure source.

   b. When the experiment is complete, the temperature should be set back to 25°C (ambient temperature) before attempting to relieve the pressure in the cell.

c. Once the cell has cooled, set the three-way valve on the manifold to the "Pressure Relief" position (arrow pointing upward). Note that the pressure cell may reduce very slowly, due to the snubbers in the manifold.

NOTE: There are two snubbers in the manifold, one of which is located on the cell itself and the other in the vent line, as described earlier in this chapter. These are used to slow the build and relief of pressure in or from the cell. The snubbers may become clogged with sample, preventing the cell pressure from being properly relieved (this problem is more likely to occur when external pressurization is being used). You must maintain the cleanliness of the snubbers, ensuring that gas can pass through them. You can verify that they are operating properly by pressurizing and depressurizing the cell without a sample. If there is any doubt concerning the cleanliness or operation of the snubbers, they should be replaced.

NOTE: The pressure relief valve on the Pressure Gauge Port on the pressure cell can also be used to relieve the pressure in the cell. This should only be done if the 3-way valve on the manifold cannot be used, if, for example, the snubbers have become blocked. The cell should also be at room temperature before using this valve, and appropriate gloves and safety glasses should be worn.

The pressure gauge located on the Pressure Gauge Port and the pressure gauge on the manifold can be used to help verify that the snubbers are not clogged. After relieving the pressure in the cell using the manifold and three-way valve, the two gauges should read zero (0) pressure. If the snubbers are clogged, the pressure gauge in the Pressure Gauge Port may still register pressure. If that condition occurs, use the relief valve on the Pressure Gauge Port to release pressure from the cell.

CAUTION: Although the pressure gauge on the cell may to appear to register zero, the level of pressure in the cell may still be above ambient.

- Disconnect the pressure cell from the manifold at the designated disconnection point at bottom of the angled pipe.

- Raise the head and decouple the magnet assembly and the rotor, and remove the Peltier jacket from the rheometer using the procedures outlined previously in "Step 4: Loading a Sample."

- Remove the cup and rotor for cleaning. See "Maintaining the Cell" for details.

Maintaining the Cell

WARNING: Before removing the heating jacket from the rheometer or the cell from the jacket, ensure that cell and manifold are not under pressure, and that the both are at safe touching temperature. The pressure relief procedure is described above.

Cleaning the Pressure Cell Cup

The cup can be cleaned simply by washing it in solvent or other type of cleaner (water, etc.), depending on the material being test, after removal from the heating jacket.

Cleaning the Rotor Assembly

CAUTION: When cleaning the rotor, observe the following precautions:

(1) Do not apply a sideways force to the rotor. Doing so can bend, break, or otherwise damage it. If a bent rotor shaft is suspected (will not rotate freely), hold the rotor assembly horizontally by the upper body (do not hold by the rotor), and spin the rotor while observing the edge. No discernible wobble should be seen as the edge of the rotor rotates.

(2) Do not allow sample or solvent to flow upward into the upper portion of the body of the rotor assembly, as this may result in a change in the friction of the sapphire bearings.

If sample fluid has flowed into the sapphire bearings and magnet of the rotor assembly, the rotor may become "sticky" (not rotate freely). In this case, the rotor assembly must be dismantled and cleaned as follows:

1. Hold the rotor assembly by the upper portion only. Be careful not to bend the rotor shaft—see Caution #1 above.

2. Place the tips (bent at 90°) of the supplied pliers into the holes of the locking nut and unscrew the nut by rotating counterclockwise (anticlockwise).

3. Remove all inner components by gently pulling the rotor straight out of the assembly. Clean the inner components using an appropriate solvent. Cellophane tape can be used to clean surfaces that have been contaminated by solid particles, but be certain that no adhesive residue remains on the components.

4. If sample has flowed to the upper sapphire bearing, remove the thumbscrew and clean the inner components using solvent.

5. Reassemble the pieces in the reverse order, being careful not to overtighten the locking nut (hand-tight is sufficient).

NOTE: Typically, sapphire bearings are designed to work "dry." The Pressure Cell is therefore supplied without lubricant. However, it has been found that a small amount of suitably compatible grease applied to the bearings can reduce the risk of a "sticky" bearing following contamination of the bearing by condensation or overflow of sample fluid.

Disassembling the Rotor

1. Insert the wrench (also called a Tommy bar) provided into one of the six holes in the rotor assembly cap and turn the rotor assembly counterclockwise until the entire assembly can be removed.

2. Place the rotor assembly upside down on a flat surface (table or lab bench). Be careful not to bend the rotor shaft—see Caution #1 on the previous page.

3. Place 90° pliers into the holes of the locking nut and unscrew the locking nut by rotating it counterclockwise (anticlockwise). See the figure to the right. (You may find it easier to holdthe locking nut steady with the pliers and rotate the rotor cap clockwise.)

4. Remove the cap covering the magnet (see the figure to the right for reference, if needed).

5. Before disassembling the remaining parts, note the scribe lines on the magnet and bob collar. This will allow you to realign the magnet properly after cleaning. See the figure to the right.

6. Continue to remove parts only if you have a torque wrench to retighten the rotor setscrews again as directed in step 13 on the next page.

7. Loosen the two setscrews on the rotor to remove the rotor from the shaft. The locking nut and sapphire bearing holder can now be removed from the shaft. See the figure below for reference.

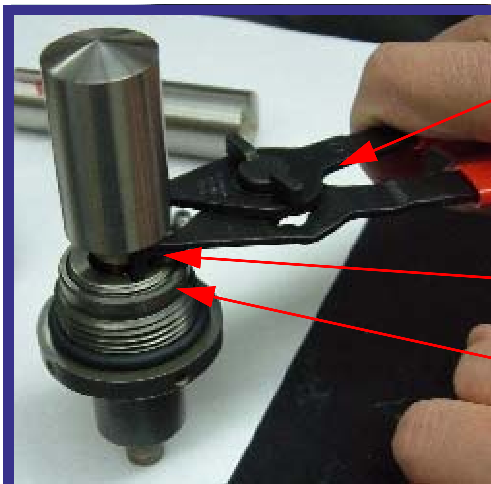


Figure 8.23
Taking Apart the Rotor Cell Assembly

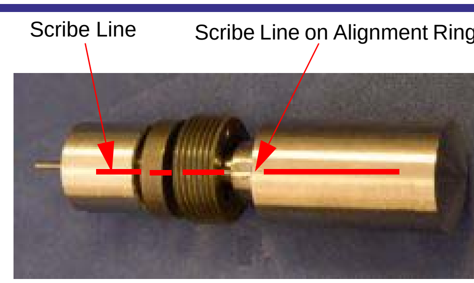


Figure 8.24
Marking Magnet to Line Up with Alignment Hole

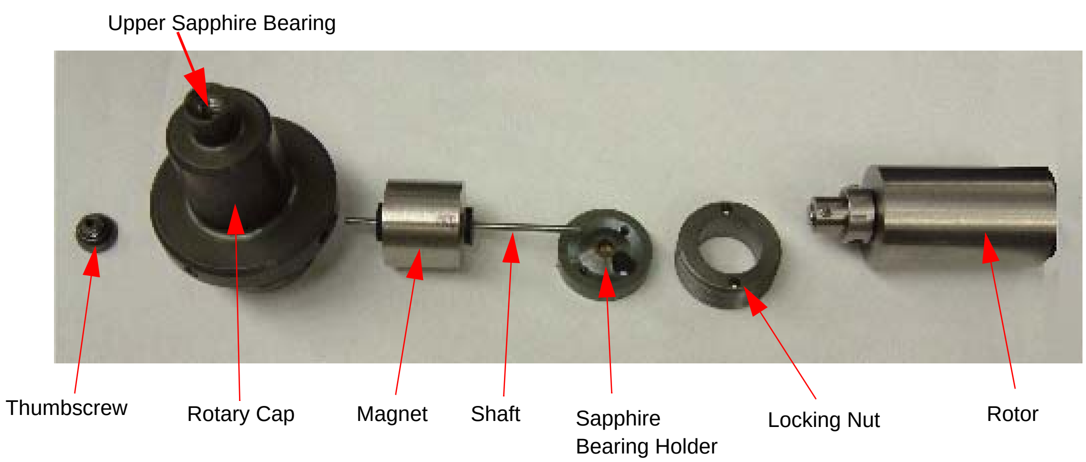


Figure 8.25
Parts of the Rotary Assembly (In correct order of assembly)

8. Clean the inner components using an appropriate solvent (*e.g.*, alcohol, acetone). Cellophane tape can be used to clean magnet surfaces that have been contaminated by solid particles, but be certain that no adhesive residue remains on the components.

9. If sample has flowed to the upper sapphire bearing, remove the thumbscrew and clean the inner components using solvent. A pipe cleaner can be used to clean inside surfaces.

Reassembling the Rotor

Make sure all of the parts are dry after the cleaning process and follow the steps below to reassemble the pieces.

10. Slide the sapphire bearing holder onto the magnet shaft making sure the flat side is facing towards the rotor (which will be on the bottom).

11. Slide the locking nut over the shaft with the plier holes facing toware the rotor.

12. Align the magnet's mark with the rotor alignment ring, insert the shaft into the rotor, and make sure the shaft bottoms out in the rotor.

13. Tighten the two setscrews on the rotor, using a torque wrench to adjust the setscrews to 8.5 lbf-in or .96 N-m.

14. Turn the rotary cap upside down and rest it on a flat surface (table or lab bench).

15. Taking the parts already loosely assembled, insert the shaft into the rotary cap until you feel it hit the bottom. You may need to move it around slightly until it drops all the way through. The tip of the shaft should be visible at the top of the rotor as seen in the figure to the right.

16. Using the 90°pliers, screw down the locking nut by rotating it clockwise. See the lower right-hand figure. (You may find it easier to tighten the nut by rotating the rotary cap counterclockwise and holding the locking nut steady with the pliers.)

17. Watch the top surface of the locking nut as it is tightened. Once the locking nut's top surface is even with the cap, the shaft must be fully inserted in the sapphire bearing as directed in step 15.

    The rotor will rest on top of the 90° pliers if the shaft is correctly located inside the sapphire bearing.

    DO NOT CONTINUE TIGHTENING UNTIL THIS IS VERIFIED. THE BEARINGS MAY BE DAMAGED IF THE SHAFT IS NOT INSIDE BOTH BEARINGS BEFORE TIGHTENING COMPLETELY.

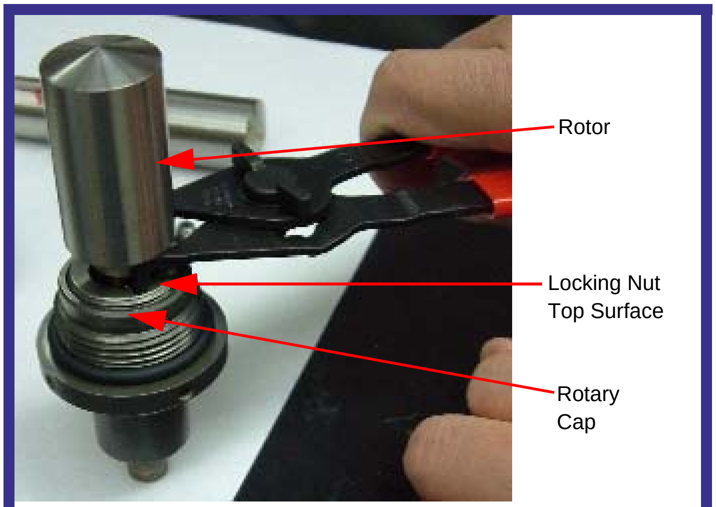


Figure 8.26
Reassembling the Rotary Assembly

18. Replace the thumbscrew at the top of the rotary cap.

19. Turn the entire assembly right-side up and place it back into the pressure cell cup.

20. Insert the wrench (also called a Tommy bar) provided into one of the six holes in the rotor assembly cap and turn the rotor assembly clockwise threading it down and into the cup.

Replacement Parts

Replacement parts for the AR Pressure Cell are available from TA Instruments. See the table below when ordering parts.

Part Number	Description
200352.002	Pressure snubber used in manifold kit
200353.001	Three-way ball valve used in manifold kit
200380.001	Wrench Open-End 7/16 & 1/2
200380.002	Wrench Open-End 7/16 & 9/16
200380.003	Wrench Open-End 9/16 & 11/16
400.09199	Wrench (Tommy Bar) used to torque the Rotor Cap into the Pressure Cell Cup
403001.901	Pressure Cell Assembly
403008.901	Upper Pressure Cell Assembly (with bob and magnet)
403029.901	Manifold Assembly
403032.901	Alignment Magnet used to hold the Rotor Assembly Magnet in place
403040.001	Sorbothane Pad used to mount the Tubing Manifold
403067.901	Safety Pressure Relief Port (with 2500 PSI Rupture Disc)
603.03519	Large Kalrez O-Ring 1.296 I.D. used between Rotor Cap and the Pressure Cell Cup
603.03557	Kalrez O-Ring 0.208 I.D. used with Thumbscrew on top of Rotor Assembly Cap
613.03378	Metric Hex Wrench Set (5, 4, 3, 2.5, 2, 1.5 mm sizes)
613.04701	90-degree Angle Pliers used on the Rotor Assembly Locking Nut

AR 2000 Interfacial Accessory

Overview

At the interface between two immiscible liquids, or between a liquid and a gas, a two-dimensional phase exists that has its own rheological properties, distinct from those of the two bulk phases [1]. Several methods of investigating the rheology of this interfacial phase have been developed [2]. One of these methods is to use a two-dimensional analogue of the standard concentric cylinder system, with a rotational rheometer [3]. Although the principles of this method were first described some years ago, it is only recently that commercially available rotational rheometers have become sufficiently sensitive to allow it to be generally used. TA Instruments has designed an interfacial accessory for use with the AR 2000 rotational rheometer and Smart Swap™ connector, which operates on these principles.

The interfacial accessory consists of a circular cup with removable lid and a thin, biconical disc geometry (Figure 1). For chemical inertness, and to reduce the meniscus effect, the cup and lid are constructed from poly(tetrafluoroethene), PTFE, and the geometry from stainless steel. It is important that the cup and disc are aligned concentrically, and base with Smart SwapÔ connection into which the cup sits has been designed to ensure this. Normally, the cup should be exactly half filled with the more dense sample fluid, and filled to the top with the less dense fluid. The disc is placed at the interface of the two fluids. A mark has been lightly inscribed on the inside of the cup to indicate when it is half full.

Figure 9.1 below shows a schematic of TA Instruments Interfacial Rheology Accessory. Liquid A is the more dense fluid, Liquid B the less dense fluid, R_D is the disc radius, R_C is the cup inner radius, H_1 is the lower fluid depth, and H_2 is the upper fluid depth. For correct operation H_1 should equal H_2.

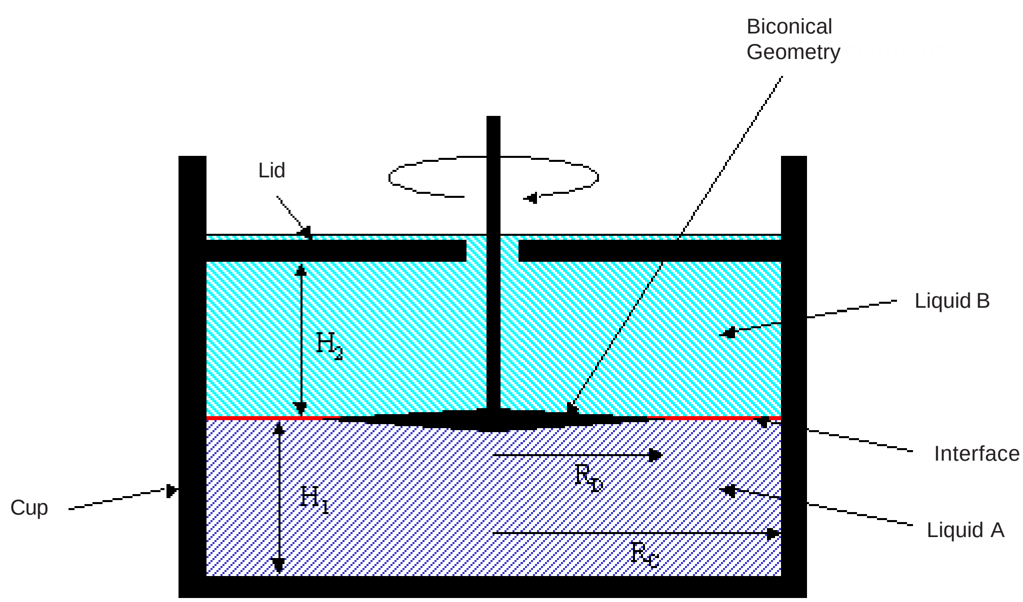


Figure 9.1
Interfacial Accessory Schematic

Specifications

Cup:	Depth	45 mm (1.77 in)
	Inner radius	40 mm (1.57 in)
	Volume	226 mL (approx)
	Material	PTFE
Geometry:	Disc radius	34 mm (1.43 in)
	Bicone angle	10°
	Inertia	20.3 mNm s2 (approx)
	Material	BS970-303 S31 grade stainless steel
Geometry gap with disc edge level with the cup half full mark (22.5 mm):		19,500 µm (approx)
Temperature Control:		Ambient only

Setting up the Interfacial Accessory

Follow the instructions below to set up the AR 2000 with the interfacial accessory:

1. Raise the instrument head, and attach the cup holder base to the rheometer using the Smart Swap™ connector, as shown in Figure 9.2 to the right.

2. Remove the lid from the cup.

3. Place the cup in the holder.

NOTE: Use the knurled grips to handle the lid. Avoid touching the under surface; these can either be gripped manually or by using a pair of fine-nosed pliers.

Figure 9.2
Interfacial Accessory Cup Holder Base

4. Thread the geometry shaft up through the hole in the cup lid and attach the geometry to the rheometer spindle.

5. Lift the lid and fix in the upper position, as shown in Figure 9.3, by passing the slots in the lid over the lugs attached to the geometry shaft, and rotating the lid.

Figure 9.3
Interfacial Accessory
Instrument Head and Lid in the UP Position (left) and DOWN Position (right)

Calibration and Mapping

Zeroing the Gap

Follow these steps to zero the gap:

1. Lower the instrument head until the bicone is within the cup, but is clear of the cup lower surface.
2. Lower the lid to sit in the groove on top of the cup as shown in Figure 9.3 on the previous page.
3. Zero the geometry gap in the usual way.

NOTE: At the zero position, the lugs on the geometry shaft will be approximately 2 mm clear of the lid.

Mapping and Other Calibrations

Mapping and other calibrations (such as for geometry inertia and bearing friction), are best carried out at this stage. Set a gap of 19500 µm, and perform the mapping and calibrations in the usual way.

Experimental Procedure

The calculation of the interfacial viscosity for the general case described previously is complicated, and can only be solved using numerical procedures [2]. However, if the first order assumption is made that the contributions from the three phases are independent of each other, the calculation becomes relatively straightforward. The contributions from each of the two bulk fluids can be obtained separately and the interfacial contribution can be obtained by subtraction of these from the total contribution.

- For this procedure, the cup is filled completely with one of the fluids and the geometry is set to gap of 19,500 µm, so that the disc edge is level with the half full mark.
- The instrument torque is determined over the range of angular velocities (or shear rates) of interest.
- The process is repeated for the other fluid, and the contributions to the torque from each fluid at each angular velocity are added.

This total will be twice that of the upper and lower fluids combined when they each occupy half the cup volume, and can therefore be halved and subtracted from the total torque, obtained in the presence of the interface, to give the contribution due to the interface. A two-dimensional analog of the concentric cylinder geometry is used to give the interfacial viscosity. Details of the analysis are given below.

Determining Each Fluid's Contribution

Follow the steps below to determine the contribution from each bulk fluid.

1. Make sure the gap is set at 19500 µm.
2. Fill the cup with Sample Fluid A, until the fluid just reaches the lower edge of the groove on top of the cup.
3. Gently lower the lid. The fluid should overflow from the annular gap between the lid and the geometry.
4. Use a Flow procedure to apply the required angular velocity (or shear rate) or range of velocities. Alternatively, apply the required torque or range of torques. It is usually preferable to use a Steady State Flow procedure for this, but the details will depend on the sample, and the reasons for conducting the experiment. This procedure gives the torque contribution of Fluid A, $M_{Acalibration}(\Omega)$, at angular velocity Ω.

   NOTE: When setting up the procedure, in the Conditioning step on the **Settings** tab, uncheck the "Wait for correct temperature" box. Any temperature settings in the procedure will be ignored.

5. After running the procedure, raise the instrument head.
6. Remove, clean and replace the cup.
7. Repeat the procedure for the Sample Fluid B, to obtain $M_{Bcalibration}(\Omega)$ then remove, clean and replace the cup.

Finding the Interface Position

Use the following procedure to find the interface position:

1. Raise the instrument head to the backoff distance then lift and fix the lid.
2. Fill the cup to the half full mark with the more dense of the sample fluids.
3. Set a gap of 24000 μm and lower the lid.
4. Ensure that the "Zero normal force before run" box is checked under **Options/Experiment/Pre-experiment**.
5. Use a squeeze test similar to that shown below in Figure 9.4. This can be set up using **Procedure/New/Flow**.

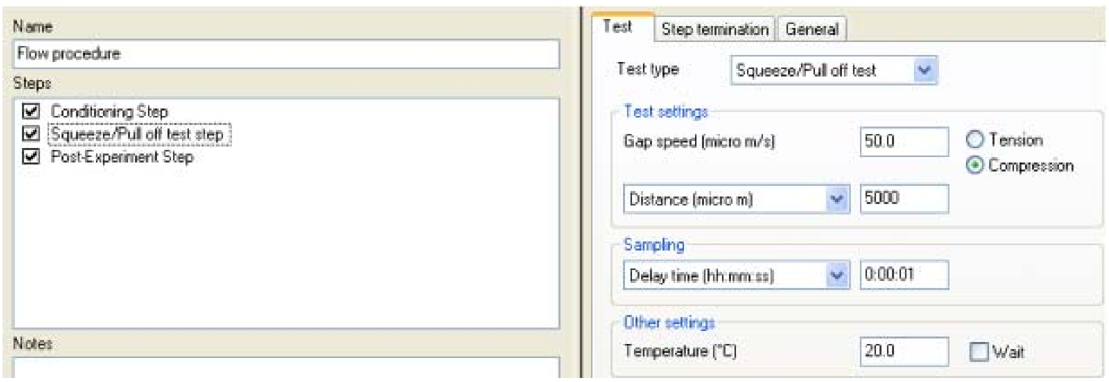


Figure 9.4: Procedure to Identify Interface Position

6. Plot the results as normal force versus gap. When the geometry is at the interface, the normal force passes through zero. To establish this point, the graph grid and cursors can be used, as shown in Figure 9.5.

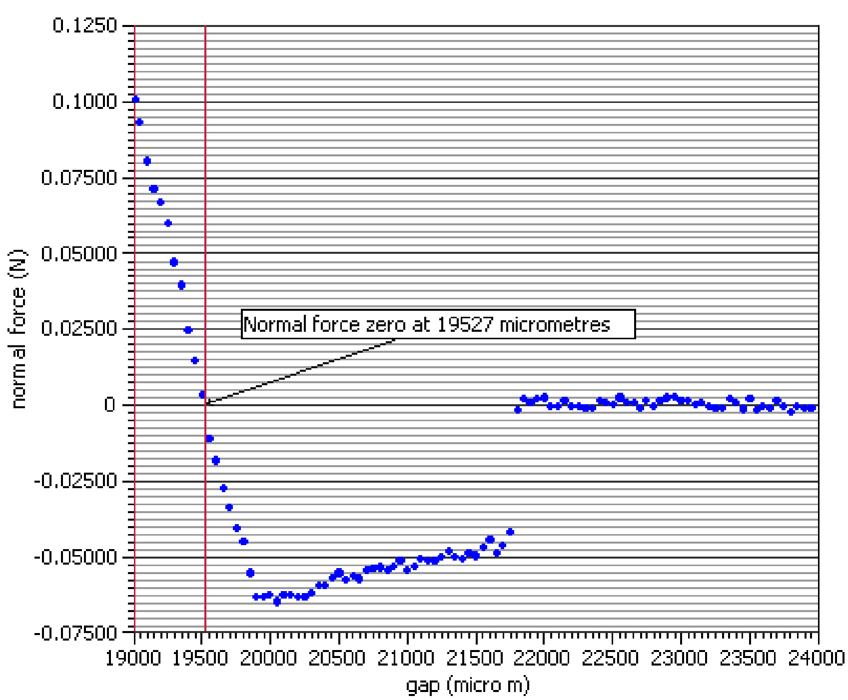


Figure 9.5
Graph to Identify the Interface Position

7. Determine the point at which the normal force passes through zero. This is the position of the interface, which should be at a gap of approximately 19500 μm. Adjust the fluid volume to obtain this gap. Keep in mind that 1 mL of fluid changes the level by about 0.275 mm.

8. When the interfacial position has been identified, manually set the gap to that position (in the example shown on the previous page, to 19527 μm).

9. Lift and fix the lid. You may find it convenient at this stage to add any surfactants to be used.

10. After addition of surfactant, gently add the less dense fluid until the lower edge of the groove on the top of the cup is just covered, then lower the lid gently. The upper fluid should just enter the annular gap between the lid and the geometry shaft. The viscosity of the total system including the interface can now be measured.

11. Run the Flow procedure used to determine the contributions of the two bulk fluids, described above, to obtain the total torque $M_{total}(\Omega)$.

Analyzing the Results

Calculation of the Interfacial Contribution to the Torque

The interfacial contribution to the torque, $M_{interfacial}(\Omega)$ at a particular angular velocity is calculated by subtracting the contributions of the two bulk fluids, A and B from the total torque for the system at that angular velocity, *i.e.*:

$$M_{total}(\Omega) = M_{interfacial}(\Omega) + M_A(\Omega) + M_B(\Omega)$$

M_A and MB, are obtained from the calibration routine described above. But $M_A(\Omega)$ is half the torque obtained at Ω for Fluid A from the calibration routine, and M_B is half that obtained for Fluid B, since for the calibration routines the cell is filled with the relevant fluid, whereas for the interfacial measurement it is half filled with Fluid A and half with Fluid B, *i.e.*:

$$M_A(\Omega) = M_{Acalibration}(\Omega) / 2 \quad \text{and} \quad M_B(\Omega) = M_{Bcalibration}(\Omega) / 2$$

Three data points are therefore needed for each angular velocity used:

- the calibration data point for Fluid A
- the calibration data point for Fluid B
- the data point for the total system

Then:

$$M_{interfacial}(\Omega) = M_{total}(\Omega) - [(M_{Acalibration}(\Omega) / 2) + (M_{Bcalibration}(\Omega) / 2)]$$

$$M_{interfacial}(\Omega) = M_{total}(\Omega) - [M_{Acalibration}(\Omega) + (M_{Bcalibration}(\Omega)] / 2$$

Interfacial Shear Stress and Shear Rate Calculation

To calculate the interfacial viscosity, a two-dimensional analog of the concentric cylinder geometry is used. The interfacial shear stress, shear rate and viscosity, are then calculated as:

$$\sigma_{\text{interfacial}} = \frac{M_{\text{interfacial}}}{4\pi}\left(\frac{R_C^2 + R_D^2}{R_C^2 R_D^2}\right)$$

$$\dot{\gamma}_{\text{interfacial}} = \Omega\left(\frac{R_C^2 + R_D^2}{R_C^2 - R_D^2}\right)$$

$$\eta_{\text{interfacial}} = \frac{M_{\text{interfacial}}}{4\pi\Omega}\left(\frac{R_C^2 - R_D^2}{R_C^2 R_D^2}\right)$$

where R_D is the disc radius and R_C the cup inner radius. For the dimensions given above:

$\sigma_{\text{interfacial}} = 118.57 \times M_{\text{interfacial}}$; $\dot{\gamma}_{\text{interfacial}} = 6.207 \times \Omega$ and $\eta_{\text{interfacial}} = 19.103 \times M_{\text{interfacial}} / \Omega$

where the units of torque are in Nm, and the units of angular velocity are in rad s^{-1}.

Note: the units of interfacial shear stress in the S.I. system are N m^{-1}, the units of interfacial shear rate are s^{-1} and the units of interfacial viscosity are Pa.s.m (pascal second metres).

Part Numbers

For re-ordering, the following part numbers should be used.

Item	Part Number
Interfacial Assembly (complete system)	545901.901
Interfacial Cup	545922.001
Interfacial Geometry	545931.001

References

[1] D.A. Edwards, H. Brenner and D.T. Wasan "Interfacial Transport Processes and Rheology" Butterworth-Heinemann, Boston (1991).

[2] S-G. Oh and J.C. Slattery, Journal of Colloid and Interface Science, 67, 516 - 525 (1978).

[3] E. Shotton, K. Wibberley, B. Warburton, S.S. Davis and P.L. Finlay, Rheologica Acta, 10, 142-152 (1971).

Chapter 10

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 in 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 μNm 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.

$$inertia \quad {}^{1}\!/_{10} \quad r^5$$

r radius
α angle of cone (degrees)
ρ density

Cylinder

Axis through center of cylinder.

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

h height of cylinder

The approximate densities (x 10^9 $\mu Nm^{-4}s^2$) 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.

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

Parameter	*Symbol*	*CGS Unit*	*SI Unit*	*Conversion CGS to SI*
Angular Displacement	ω	rad	rad	No change
Angular Velocity	ϖ	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	

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