

AR Rheometer Instrumentation, Calibration & Maintenance



2008 TA Instruments



Agenda

Instrumentation Calibration Maintenance



Agenda

► Instrumentation

Calibration





Make sure that Air is on





Unscrew Draw-rod to release bearing lock





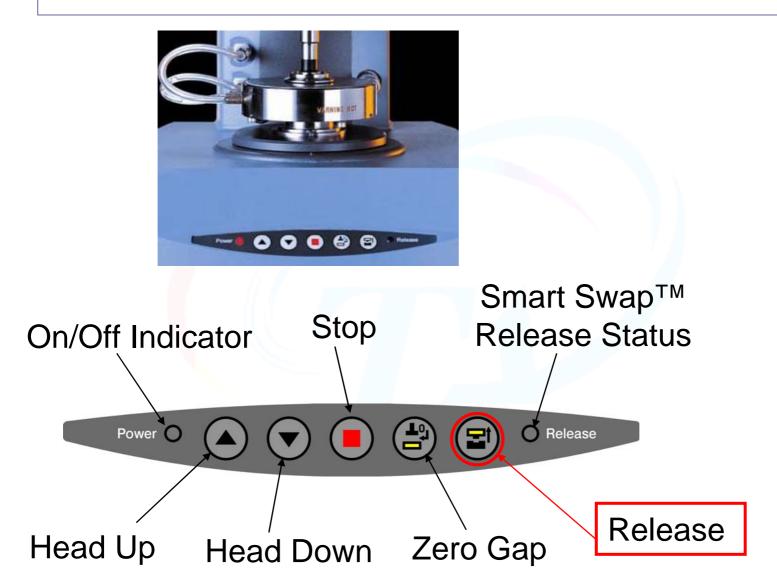






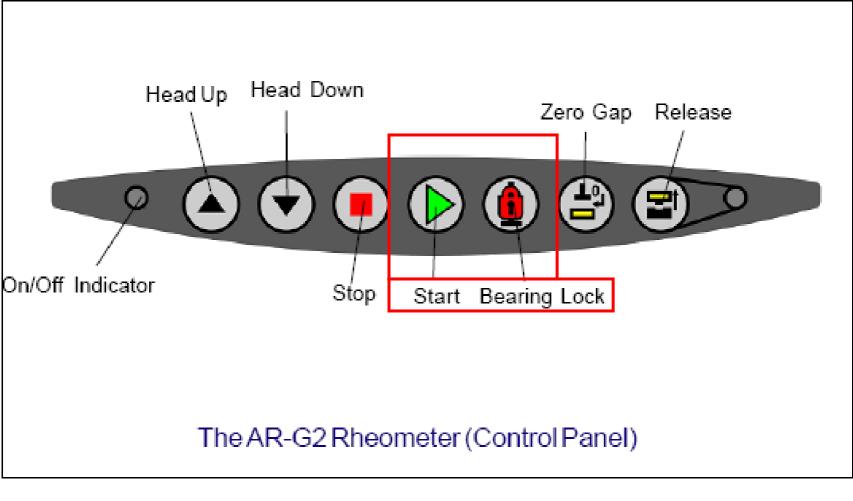
Turn on Water (Pump) (if needed)

AR 2000 Keypad





AR G2 Keypad





Peltier Plate

A device that actively heats and cools using reverse thermocouple junctions

- Best suited for dispersions, gels and solutions
- Heating from one direction
 - Should allow sample to equilibrate for at least 3 min.



Environmental system

The Peltier plate

The Peltier plate system is mounted directly to the smart swap base of the AR2000 and AR-G2. An upper heated compensation heater [UHP]is available to eliminate any vertical temperature gradients in the sample.

The Peltier plate has a temperature range from -20°C to 200°C. A humidity cover to prevent evaporation of solvent is available also.





Peltier Plate

Works as a Heat Pump

Needs a heat sink – usually water, either from a reservoir or a flowing supply

Flow rate of fluid needs to be at least 0.5 L/min

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

Concentric Cylinders

Low viscosity materials ♦ < 50 cP, 0.05 Pa-s</p> Sample volume crucial A good way to load is measuring the volume suggested in the geometry page

Peltier heating & cooling on AR 2000 & G2



🤹 RI	eology Advantage Instrument Control AR - [ArGeom2]	
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	AR2000 L Standard-size DIN or conical Concentric cylinders	ArResults-0001f)
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Environmental Test Chamber (ETC)

- Provides temperature control for polymer melts, and solid samples
- Lower plate assembly is attached and detached just like the Peltier plate



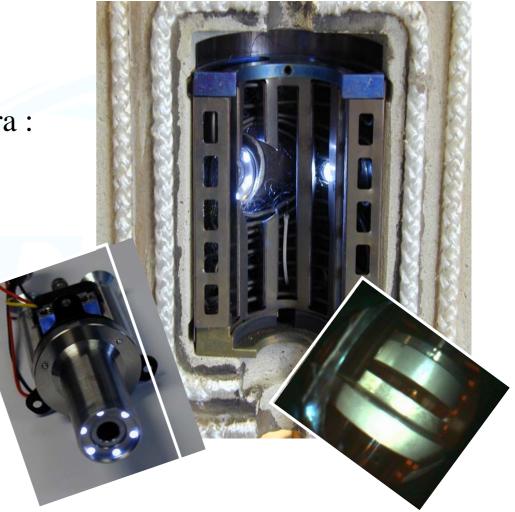


Environmental system – AR-G2

The radiation oven

The radiation oven has been enhanced with a video camera :

- Allows the observation of the sample during sample loading and the measurement
- Stores an image with each single data point to keep a record of the sample's state during the measurement



Press 'Release' button



Continuous green status light indicates attachment can be fitted...



...and plugged in





SmartSwap Cable

When green status light goes out, system is ready for use



Removing the Peltier Plate



Press 'Release' button

Flashing green status light indicates it is safe to unplug



Removing the Peltier Plate



Press 'Release' button again

Continuous green status light indicates attachment can be removed



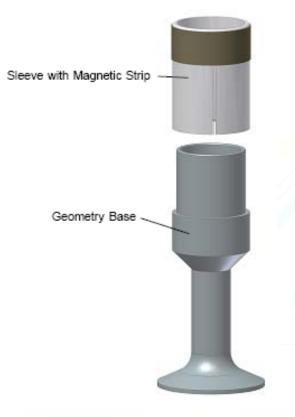


Geometries

- To ensure that data is correct you must correctly describe and choose the geometry
- On AR2000 and older Rheometers this means you have to go in and select from the software
- On AR-G2 we have new Smart Swap™ geometries, that will automatically be recognized

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Smart Swap Sleeve -- AR-G2



Automated system configuration due to smart swap for the environmental systems and the upper geometries



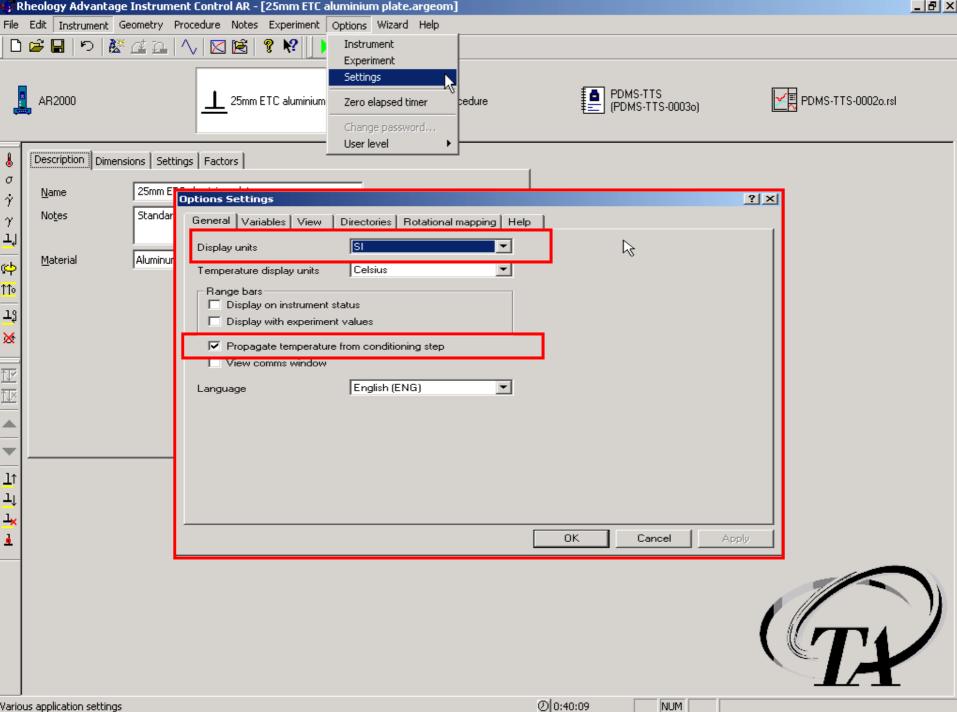
Smart swap geometry changed

The instrument is reading the smart swap geometry serial number.

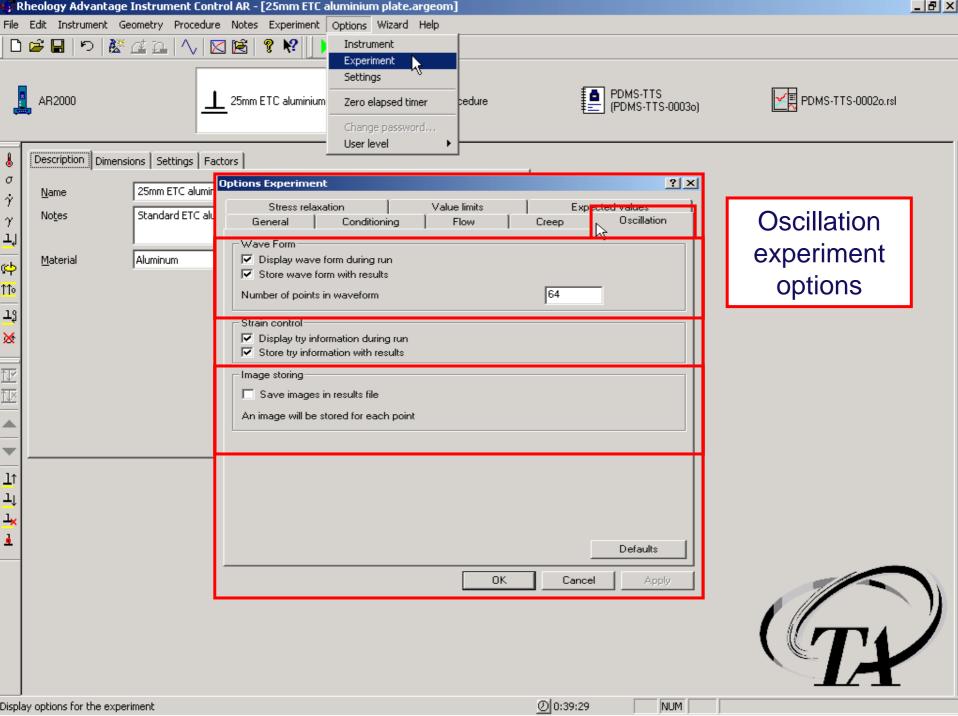


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User level	<u> </u>		
σ Name 25mm ETC aluminium plate			
γ Notes Standard ETC aluminium parallel plate μ		Let's take a quick look at	
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AR2000	25mm ETC aluminium Zero elapsed timer pedure PDMS-TTS (PDMS-TTS-0003o)	o.rsl
	User level	
Bescription Dimensions Settings Fa	actors	
σ <u>Name</u> 25mm ETC alumin	Options Experiment	
γ – Note: Standard ETC all	Stress relaxation Value limits Expected values	
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<u>φ</u>	Collect all points, flagging as invalid if below minimum velocity	
<u>^</u>	Collect negative shear rate data if stress is positive	
12 C	Zero strain at the start of each flow step	
₩	✓ Inertia correction	
	Steady state flow point graph	
	Display point graph during run	
	Store point graph with results	
▲	Image storing	
▼	Save images in results file	
11	Store an image at the next point after every n seconds 10	
1) 1)	Note: The above settings do not apply to stepped or steady state tests where an image is stored for each point.	
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	Defaults	
	OK Cancel Apply	
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Calibrations

There are two instrument calibrations that are recommended at least <u>once a month</u>

- Instrument Inertia
- Bearing Friction Correction
- A couple more that are recommended from time to time
 - Geometry Inertia
 - Gap Temperature Compensation
- Not really a calibration but should be done regularly
 - Mapping



What is Inertia?

Definition: That property of matter which manifests itself as a resistance to any change in momentum of a body

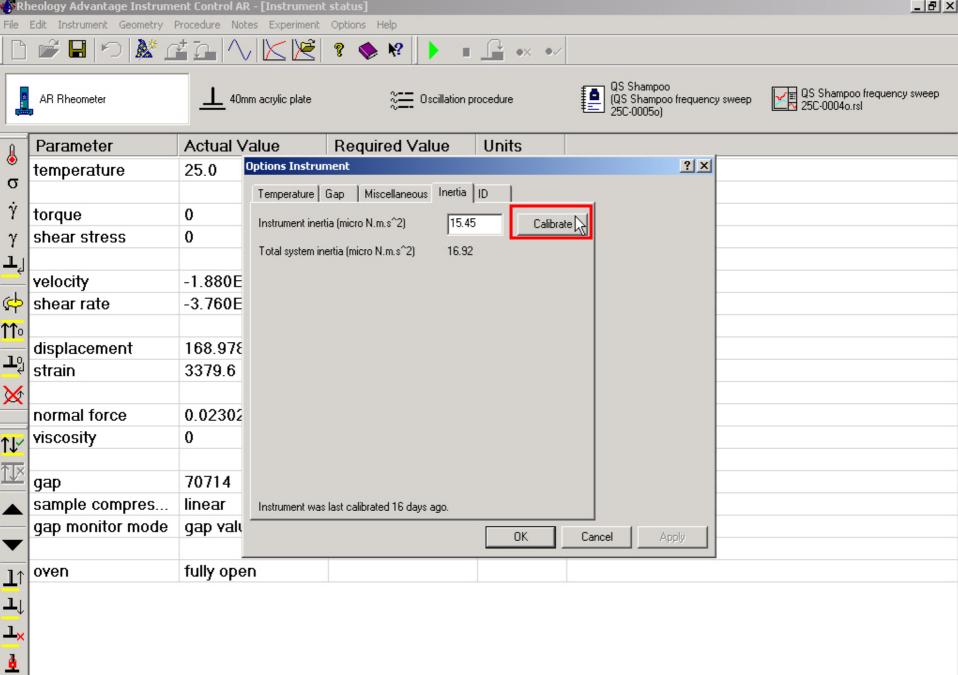
- A couple of facts
 - All motors have inertia
 - All rheometers have motors, so

We can measure the inertia of the instrument and the system inertia – instrument + geometry

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σ			Temperature	Gap Miscellaneous I	nertia, ID			
Ϋ́	torque	0	Current temp	erature system Peltier p	blate			
γ	shear stress	0		ture control enabled				
⊥ ,				perature compensation				
	velocity	7.350E-		s only (no active cooling)				
¢	shear rate	1.470E-	- Temperature	calibration				
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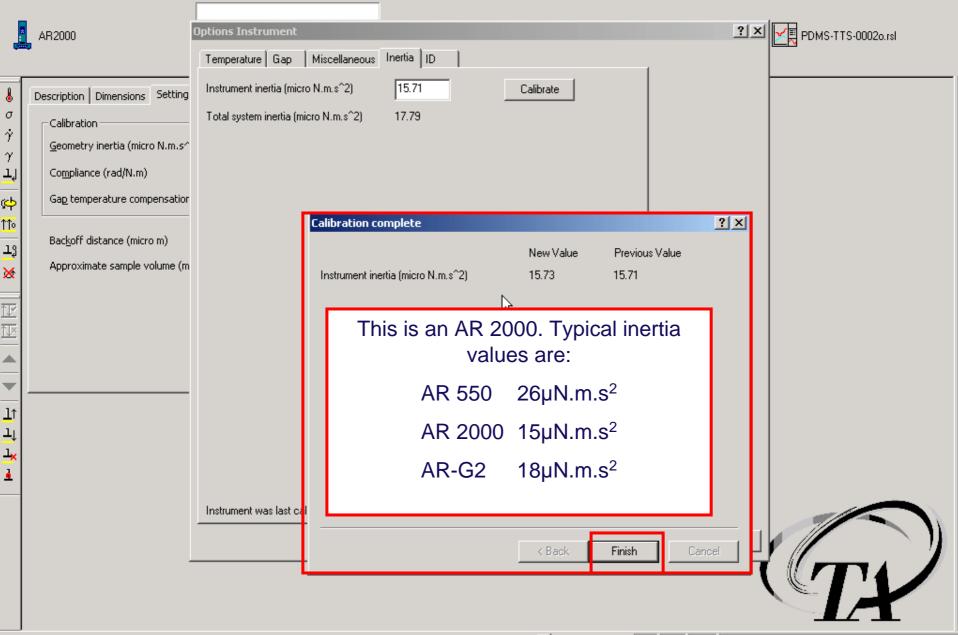
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Rheology Advantage Instrument Control AR - [25mm ETC aluminium plate.argeom]

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Calibrations

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Bearing Friction

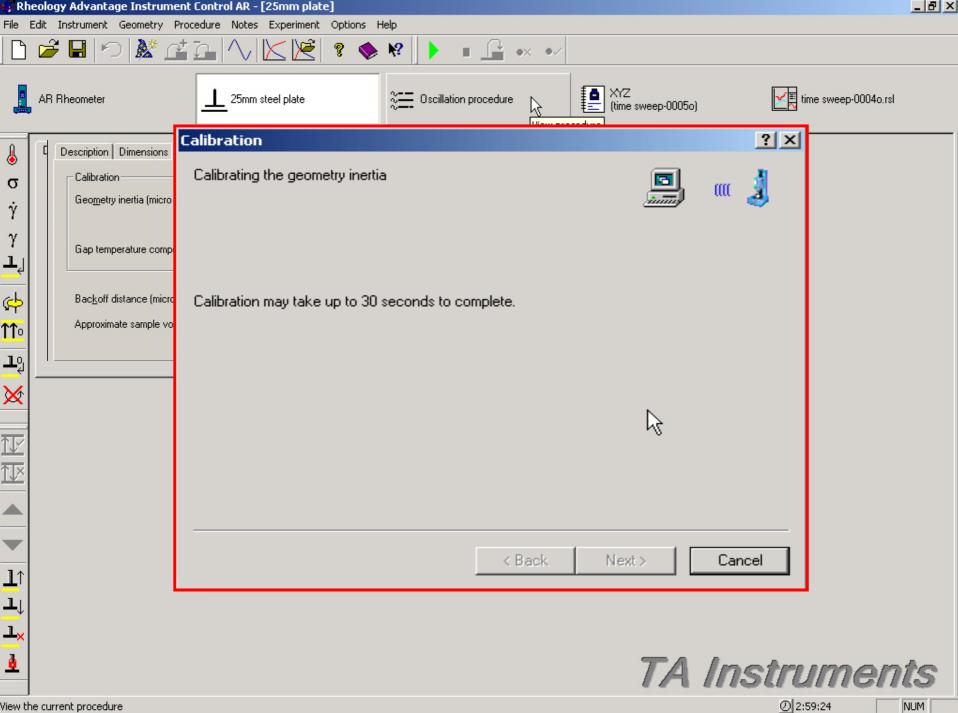
- Nobody has created a frictionless bearing
- The lower the friction, the better the low torque performance
- Bearing friction can vary depending on gap of thrust bearing and type of bearing

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	temperature	25.0	Options Instru	nent				? ×	
σ			Temperature	Gap Miscellaneous	Inertia	ID			
Ϋ́	torque	0.06	🔽 Bearing I	friction correction					
γ	shear stress	4.934E-			0.921		NI		
⊥ ,∣				on (micro N.m/(rad/s))		Calibrate			
	velocity	-0.0671	Torque offse	t (micro N.m)	0				
¢	shear rate	-1.343	- Temperature	calibration					
<mark>11</mark> ₀			System Deliver elete		Span 1 0000	Offset (*C)			
	displacement	1124.22	Peltier plate Torsion ove	en - plate	1.0000 1.0000	0 0			
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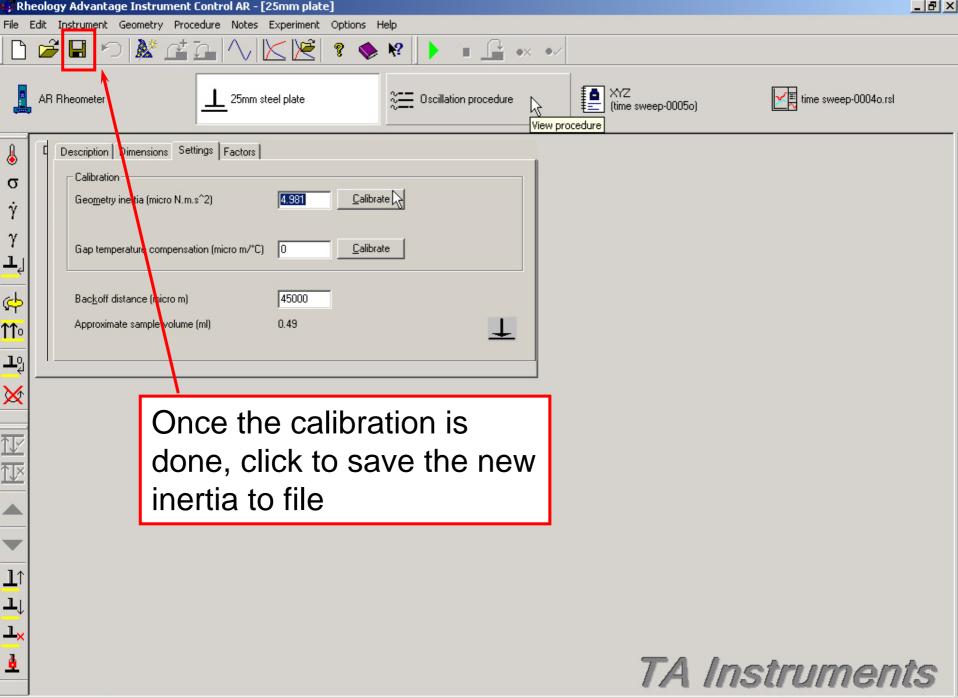
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	AR Rheometer	<u> </u>	n acrylic plate	% Oscillation) procedure	QS Shampoo (QS Shampoo frequency 25C-0005o)	y sweep QS Shampoo frequency sweep 25C-0004o.rsl
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	temperature	25.0	ptions Instru			? ×	
σ			Calibration	complete		<u>?</u> ×	
Ϋ́	torque	-0.03			New Value	Previous Value	This is for an AR
γ	shear stress	-1.989E	Bearing frict	ion (micro N.m/(rad/s))	0.920	0.921	2000. Bearing
₽Į	and a site	0.00700					friction for an AR
æb	velocity shear rate	0.02769					
¢¢		0.5556					G2 is
<mark>11</mark> ₀	displacement	1124.09					approximately
	strain	22482					0.3 – 1/3 of the
≫							
	normal force	0.02281					AR 2000 due to
î1∕	viscosity	3.592E-					the lower friction
<u>t</u> r							of the G2's
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8							as the AR 2000.

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	AR	R Rheometer	steel plate	≳==_ Oscillation proce	dure	XYZ (time sweep-0005o)	time sweep-0004o.rsl	
ס י ב_ן ג⇔		Description Dimensions Settings Factors Calibration Geometry inertia (micro N.m.s^2) Gap temperature compensation (micro m/*C Backoff distance (micro m) Approximate sample volume (ml)	4.981	Calibrate		e Geometry r <u>Settings</u>	page,	
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View the current procedure





Typical Inertia's

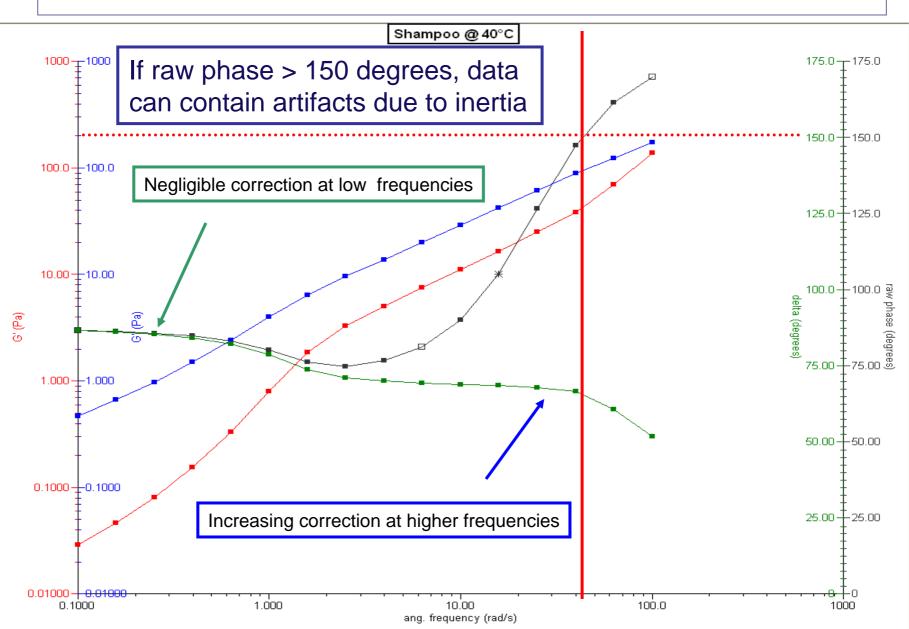
Diameter	Acrylic	Stainless Steel
(mm)		
20	0.43	2.8
40	1.34	6.92
60	3.03	23.32

System inertia in AR Rheometer

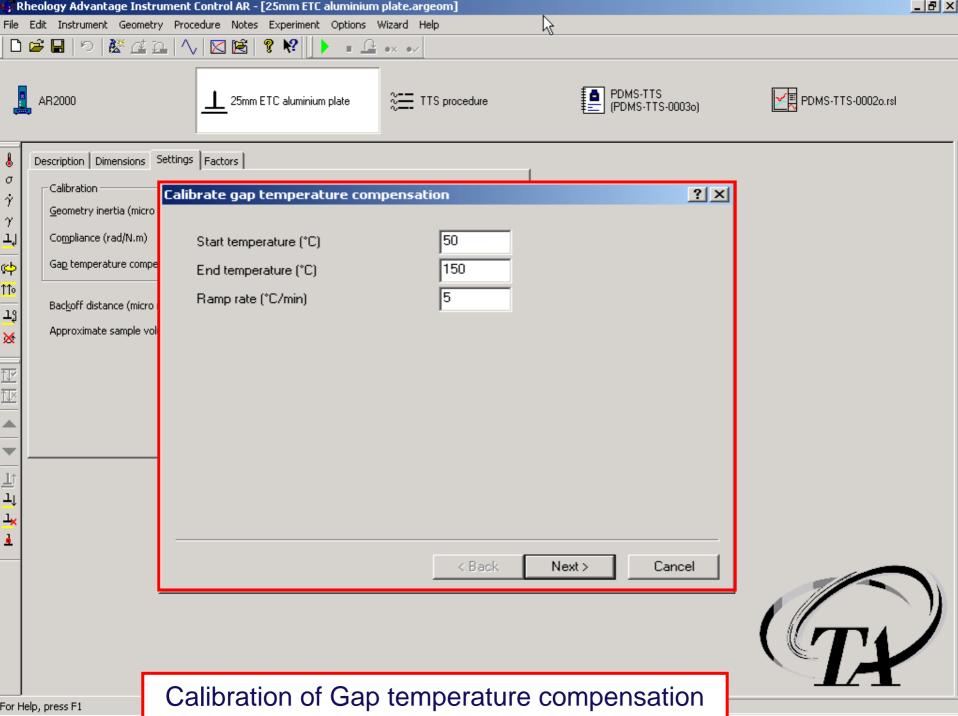
- The contribution of system inertia can be observed by plotting *Raw Phase* raw phase * Inertia Correction = delta
- Raw phase is the uncorrected phase angle for inertia

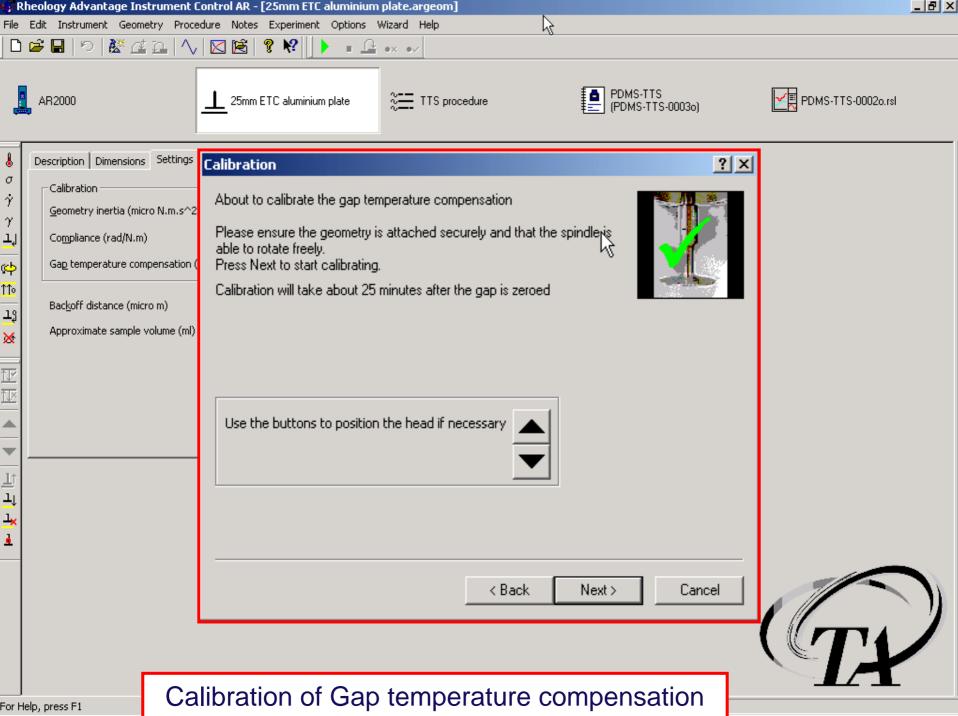
When raw phase is greater than 150 degrees, the contribution of the instrument (system inertia) in the measurement is greater than from the sample

AR Correction for Inertia

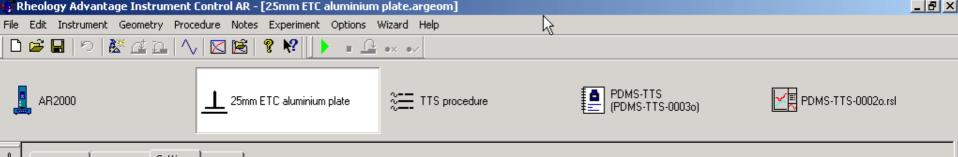


AP200 AP	🕐 Rheology Advantage Instrument Control AR - [25mm ETC aluminium plate.argeom]		_ 8 ×
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Description Dimensions Setting Factors Calibration	D 🖆 🖬 つ 🛣 🖆 🕰 🔨 🗹 🗟 🧣 😵 🕨 🗉 🕰 🐭 🛶		
Calibration of Gap Geometry Inetia (micro N.m.s^2) 2.230 _ calerate Cogpliance (rad/N.m) 3.130E-3 Geograficate (micro m) 26500 Approximate sample volume (m) 0.49 L Calibration of Gap temperature compensation	AR2000 25mm ETC aluminium plate	PDMS-TTS (PDMS-TTS-0003o)	
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δ Description Dimension σ Calibration γ Geometry inertia (m 1 Compliance (rad/N.r.)	micro N	
Gap temperature co ↑↑ ⊥3 Approximate sample	micro m	
<u>⊥</u> ⊥ 1 ×	Calibration will take about 25 minutes	
	< Back Next > Cancel	
For Help, press F1	Calibration of Gap temperature compensation	X



Description Dimensions Settings Facto	ors			
Calibration	Gap temperature compensation calibra	tion complete		<u>?</u> ×
Geometry inertia (micro N.m.s^2)		New Value	Previous Value	
Compliance (rad/N.m)	Gap temperature compensation (micro m/*C)	2.5	0	
Gap temperature compensation (micro n	Regression	0.99939		N
Bac <u>k</u> off distance (micro m)				2
Approximate sample volume (ml)				
		< Back	Finish	Cancel
o, press F1 Calib	ration of Gap temperatu	re comp	ensation	

Calibrations

There are two instrument calibrations that are recommended at least <u>once a month</u>

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Not really a calibration but should be done regularly

♦ Mapping

		Control AR - [Instrument status]			
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	AR2000	25mm ETC aluminium plate	‰ <u>——</u> TTS procedure	PDMS-TTS (PDMS-TTS-0003o)	PDMS-TTS-0002o.rsl
8	Parameter	Actual Value	Required Value	Units	
σ	temperature	21.7	80.0	°C	
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γ LJ	torque	0.04		micro N.m	
ᆛ	shear stress	0.01271	0	Pa	
¢¢,					
	velocity	-0.07185		rad/s	
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	displacement	-765.6750		rad	
<mark>≿</mark> ⊭	strain	-260.06			
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	normal force	8.895E-3		N	
▼	viscosity	-0.5209		Pa.s	
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' 	gap	36803	36803	micro m	
1ੇ ⊐ੇ <mark>1</mark> ੋ 1	sample compression	exponential			
1	gap monitor mode	gap value			
	oven	fully open			

Rotational Mapping

Rheology Advantage Instrument Control AR - [Instrument status]

File Edit Instrument Geometry Procedure Notes Experiment Options Wizard Help

•	AR2000	<u> </u>	Rotational mapping	PDMS-TTS-0002o.rsl
0	Parameter	Actua	Date: 10/6/2005 1:47:15 PM	
• σ	temperature	21.7	Geometry: 60mm Acrylic plate	
Ϋ́	·····			
γ	torque	0.04		
1,	shear stress	0.012	Mapping settings	
ф,			Bearing mapping type precision 💌	
î∘¦	velocity	-0.071	Number of iterations 2	
13 13	Instrument rotational mapping	-0.024	,	
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	normal force	8.895		
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ıţ	gap	36803		
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Mapping Types

- Standard
 - Approx 1 min to complete
- ➤ Fast
 - Approx 45 sec to complete
- Precision
 - Takes about 2 min to complete
- Extended (AR 2000 only)
 - Best low torque/velocity performance

Mapping Iterations

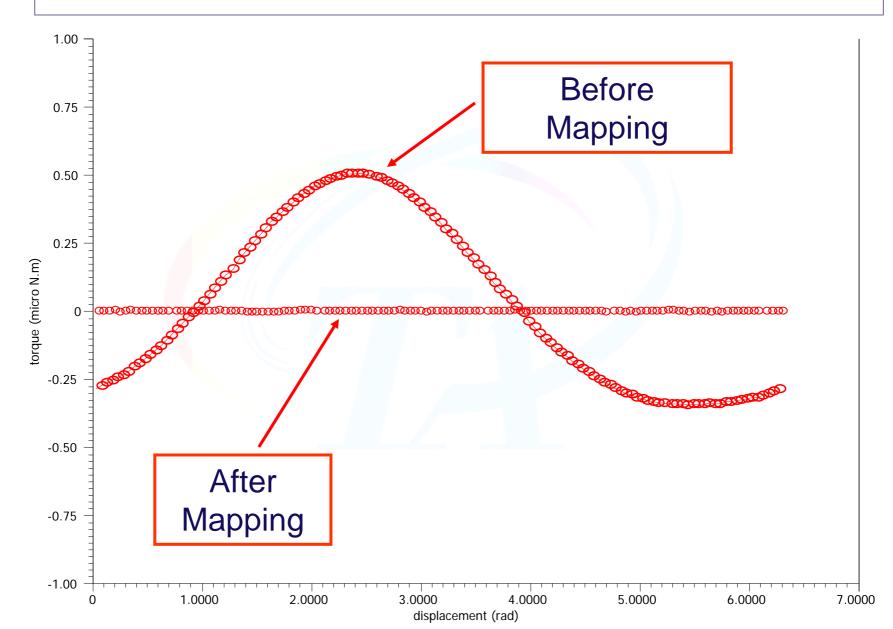
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-Last mapping -			
Date:	10/6/2005 1:47:15 PM	1	
Geometry:	60mm Acrylic plate		
-Mapping setting	js		
Bearing mappi	ng type	precision	•
Number of iter	ations	2	
Mapping may t	ake up to 5 minutes to:	complete.	

The more iterations the better to a point. Improvement is reduced after 2 iterations.

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	AR2000	<u> </u>	n ETC aluminium plate 💥 TTS procedure PDMS-TTS (PDMS-TTS-0003o)	
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ੀ ⊐੍ਰੋ <mark>ਤ</mark> ੍ਰੋ	gap	36803	36803	micro m		
	sample compression	exponential				
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Residual Torque Before & After Mapping



System Verification

- Attach a 60 mm 2° cone (if available, if not then use the largest cone available)
- > Zero
- Load sample of certified standard oil
- Carry out a 2 min flow test over as wide a range as possible
- Determine Newtonian viscosity. If it is more than 5% different from certified value repeat the experiment. If the results are still in error contact TA for advice



Sources of Error

Over or underfilling of gap

- Ensure that you have a standard method of sample loading
- Temperature error
 - Verify the temperature against a certified digital thermometer
- Gap Setting
 - Ensure that the gap was correctly zeroed and that the correct truncation is being used



Maintenance

- Check air filter and regulators for proper functioning
- If air must be turned off, then make sure that the bearing lock is fastened prior to turning air off
 - NOTE: <u>Do not</u> rotate drive-shaft if air goes off
- Confidence check: Cannon certified viscosity standards
 - 250024.001: Low viscosity standard oil
 - 250024.002: High viscosity standard oil



Check Air Filters

Air supply should be clean and dry
 Check for moisture and contaminates at least once a month





Manual & Help files in RA

- The online help manual is an excellent source of information
 - *.pdf files, with information installation, operation and some basic theory
- In the Rheology Advantage, Click on Help



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Operators Manual

Reprogramming the Serial Number

An attempt to reprogram the serial number can be made by following these steps.

 SelectOptions/Instrument/ID and click on the Service button the dialog shown to the right is displayed.

vice	
Firmware update	
Select this button to update the firmware in the instrument	Berform update
Dearing test	
	Perform test
Encoder Inearisation	
Encoder linearisation needs to be performed when the firmware is first installed. It will take about an hour to run and CANNOT be cancelled.	Perform Inearisation
Program smart swap serial number	
	Program

Select the Program button. The dialog shown below is displayed.

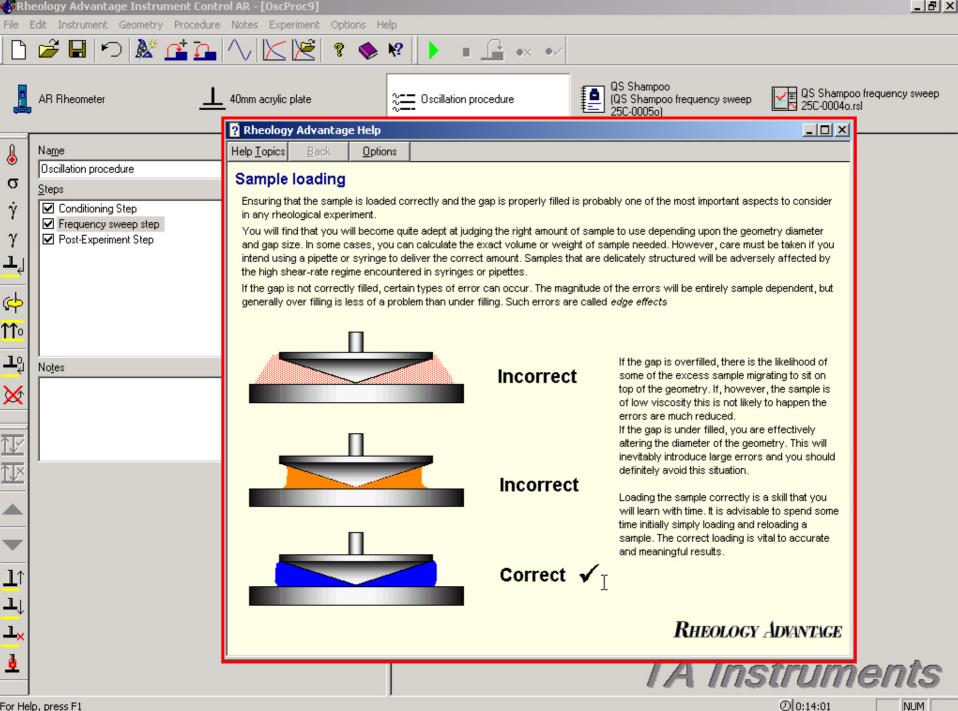
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mber (1000 - 999999)		 Select the Smart swap geometry radio button. The instrument will attempt to read the existing serial number and will return a value of 1000, if nothing car be read.
		4. Enter the serial number of the geometry (located on

From the AR-G2 manual

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	۵,	Oscillatory tests	sweep
	P te	We learn at an early age that materials may be gases, liquids or solids. But this is an oversimplification. Many industrial materials show behaviour which is neither completely liquid nor completely solid, but is somewhere between the two. Such materials are termed viscoelastic. Typical examples are polymer solutions and melts, and particulate dispersions such as paints, inks, drilling fluids, creams and lotions, and many types of foodstuffs.	
σ	tc	It is viscoelasticity which is responsible, at least in part, for the handling properties of these materials, and it is important that they should exhibit it in the correct degree. For example if a printing ink is too elastic (solid) it will fail to enter the nip, whereas if it is too liquid it will show poor dot definition. There are several ways of examining the viscoelastic properties of materials, but the commonest, and most versatile, is to use oscillatory rheology.	
Ļ	st Ye	If a sinusoidal stress, σ (force acting over an area), is placed on a <i>solid</i> sample, a sinusoidal displacement (strain, γ) will result which is in phase with the applied stress. The modulus, or stiffness, of the material can be obtained by dividing the amplitude of the stress, σ0, by the amplitude of the strain, γ0 (Figure 1):	
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⊥ º	di st	$V V V V \downarrow $ strain amplitude	
	n	Figure 1 strain response to a sinusoidally applied stress for a solid material	
1↓′ 11	Υİ	If a sinusoidal stress is applied to a <i>liquid</i> sample, the stress is in phase with the rate of change of strain, and a phase lag of 90° is therefore introduced between the stress and the strain (Figure 2):	
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4		Figure 2: strain response to a sinusoidally applied stress for a liquid material	-

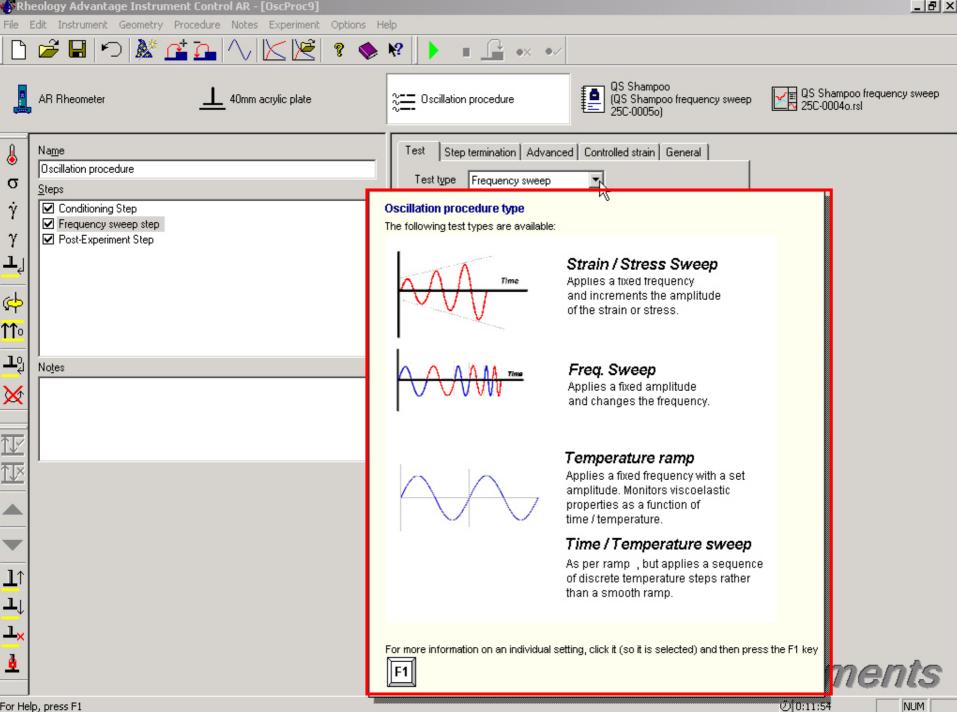


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Do

- Ensure a clean dry air supply at a stable pressure. Use a filter and dryer
- Check filter bowls for water & contaminants at least monthly
- Replace the air-bearing clamp before turning the air off
- Ensure air supply is on before turning the instrument on
- Take advantage of the training opportunities offered



Don't

- Operate instruments without air
- Touch the spindle without air being on if air is inadvertently turned off make sure power is turned off and wait till air turns back on before placing clamp on or turning power back on
- Operate without a water supply if using the Peltier
- Hesitate to contact us if you have any questions