



INTRODUCTORY GUIDE TO USING AN AR SERIES RHEOMETER USING RHEOLOGY ADVANTAGE SOFTWARE* (VERSIONS ≤ 4.1)

Fred Mazzeo, Ph.D.

TA Instruments, 109 Lukens Drive, New Castle, DE 19720, USA

1. Turn on the computer controller.
2. Turn on the air supply to the rheometer. Pressure requirements are as follows: AR2000: 30 psi, AR1000: 25 psi, AR 550/500: 37.5 psi.
3. Remove the black bearing lock by holding it in place while turning the draw rod knob at the top in a counter-clockwise direction. Once the bearing lock is removed, make sure that the spindle rotates freely.

NOTE: For an AR2000 system with a drive-shaft slide lock, please ensure that the drive-shaft slide lock is pulled-out.

NOTE: If air supply is interrupted while bearing lock is off, **DO NOT TURN the DRIVE SHAFT**, this will cause damage to the bearing. Locate another gas source, attach it to the gas port on the rheometer, and then attach bearing lock while the air bearing is floating.


4. Turn on the Power to Rheometer

Switch the power button, located on the rear of the electronics control box, to the *ON* position.

For AR1000/550/500 systems equipped with an ETC, turn on the power to the ETC electronics box.

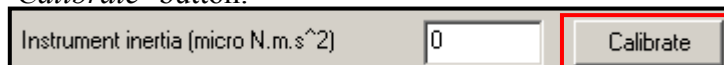
NOTE: If step 4 is performed before step 3, an alarm will sound and the instrument controller display will read ‘optical init. fail’. At this point, turn off the power and perform step 3.

5. When using a Peltier control device, please ensure that the water supply is turned on. If using a pump and tank system, ensure that the water is clean and the pump is fully submerged.
6. When the instrument has finished the system check, turn on the instrument control software: *Start>Programs>TA Rheology Advantage>AR Instrument Control*.

7. Choose the *Instrument Status Page* icon, , to make certain that communication has been established between the computer and the instrument. If communication is not established, the page will appear blank. The Instrument Status Page can also be used to manually control the instrument.

8. Instrument Inertia

Determine the instrument inertia by selecting *Options>Instrument>Inertia* and press the ‘*Calibrate*’ button.



Instrument inertia (micro N.m.s²)

This value is unique for each air bearing assembly. An acceptable range for this value is ~14-16 μNms^2 for the AR1000 and AR2000, and ~26 μNms^2 for the AR500 and AR550. The value for the instrument should not change by more than

15% of the original Inertia value. _____ ± _____

If you notice a continual drift of this value, check the quality of the air used, as it could be indicative of a poor quality supply. If the problem persists, contact the instrument service department.

9. Geometry

Attach test geometry by sliding it up the drive shaft and hold it stationary while turning the draw rod knob at the top in a clockwise direction. If the geometry file was previously created, choose the appropriate geometry (*Geometry>Open...*), or create a new geometry by selecting *Geometry>New*, and follow the New Geometry dialogue window.

NOTE: If testing a solid sample, attach the appropriate spacers based on the sample's dimensions before calibrating the geometry. Refer to 18.c. for determining spacer size.

10. Geometry Inertia

The value of the inertia for each measuring system differs because they all have been uniquely engineered and have different masses. It is important to calibrate the inertia value for every geometry, particularly if high frequency oscillations are being used, or if low viscosity fluids are being measured.

Calibrate the geometry inertia by pressing the 'Calibrate' button that is found in

the *Geometry Page*  > *Settings>Inertia: Calibrate*.



11. Bearing Friction Correction

An air bearing is used to set the drive shaft afloat and provide virtually friction free application of torque to the sample. However, there will always be some residual friction. With most test materials, this is insignificant, but in about 1% of low viscosity samples, this inherent friction causes inaccuracies in the final rheological data. To overcome this, the software has an air bearing friction correction that should be activated.

NOTE: Please ensure that the Instrument (**Step 9**) and Geometry inertia (**Step 10**) has been calibrated before determining the 'bearing friction correction' value.

If using *Rheology Advantage 3.0* or higher, go to *Options>Instrument>Miscellaneous*, check the 'bearing friction correction' box and press the 'Calibrate' button.



If using *Rheology Advantage 2.0* or lower, uncheck the bearing friction correction box located within *Options>Instrument>Miscellaneous*. Go to *Help>Index*, type in the phrase 'friction: calibration>determining bearing friction correction' and follow the steps.

This value is unique for each air bearing assembly and rheometer model. An acceptable range for this value is ~0.5-1.1 $\mu\text{Nm}/(\text{rad/s})$. This value should be within ~20% of the original BFC value. _____ ± _____

12. Temperature System Selection

For an *AR2000* system, attach the appropriate Smart Swap lower geometry temperature stage/system. (See Smart Swap operational instructions by going to *Start>Programs>TA Rheology Advantage>Rheology Advantage Manuals: AR2000* pg 5-6)

For an AR1000/550/500 system, attach the appropriate lower geometry temperature stage, not necessary if using the Peltier plate. The temperature read and control must be set to the appropriate temperature system. This is accomplished by choosing *Options>Instrument>Temperature: Temperature Read* and *Temperature Control* setting both to ‘Peltier’, if using the Peltier Plate, or ‘Temperature System’ if any other system is being used, such as a water circulator, ETC or ETM.

NOTE: If the rheometer is equipped with an ETC and it is being used for testing, set the ETC flow meter to 10 lpm.


NOTE: If using Rheology Advantage 4.0 or greater and using an ETC equipped with low temperature fittings, checking ‘purge gas only’ (located in *Options>Instrument>Temperature*) will convert a low temperature flow meter to use only the ETC purge gas instead of Liquid Nitrogen to control temperature. Recommended lower temperature range when using this setting is 45°C. For tests that require lower temperatures using ETC, attach LN₂ feed and disable this setting.

13. Mapping

a. **Rotational Mapping:** Due to the micron-level tolerances needed to make an air bearing work, any bearing will have small variations in torque behavior around one complete revolution of the shaft. They are consistent over time unless changes occur in the air bearing.

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 in memory for subsequent real-time corrections in the test. To create a map, the software rotates the drive shaft at a fixed speed, monitoring the torque required to maintain this speed through a full 360° of rotation. This results in a very wide operating range of the bearing without operator intervention - a confidence check in bearing performance.

Perform a rotational mapping on the geometry when the test procedure will be applying either a flow or transient (Creep or Stress Relaxation) mode of

deformation. Begin the rotational mapping by pressing the icon  or go to *Instrument>Rotational Mapping*.

If using Rheology Advantage 3.0 or higher, select either the number of iterations or mapping type within the icon dialog window.

If using Rheology Advantage 2.0 or lower, go to *Options>Settings>Mapping* and *Options>Instrument>Miscellaneous: Mapping Type*.


The number of points in the map and the speed of rotation used are dependent upon the mapping type used. When mapping the geometry, the recommended settings are ‘**one**’ iteration and ‘**standard**’ type.

- i. The mapping type can be set to fast, standard, precision (AR500/550/1000/2000) or extended (AR2000 only). For critical low-torque measurements, using <10 μNm, ‘precision’ mapping is more suited, unless ‘extended’ mapping is available.
- ii. If ‘extended’ mapping is available (AR2000 only) and chosen, the number of iterations is not an option. When using fast, standard or precision mapping the number of iterations should be set greater than one. If performing a Creep procedure and using fast, standard or precision mapping, the number of iterations should be set to **four**, if the Recovery step is set to **zero**. Otherwise, setting the

number of iterations greater than three has diminishing returns in the mapping performance.

b. **Oscillation Mapping** [AR2000 only, using 4.1]: This mapping will perform a baseline subtraction only when using the continuous controlled strain mode and will improve the performance for low torque, low displacement data. To access in the software, go to *Instrument>Oscillatory mapping*. Refer to *TA Rheology Manuals* found on the computer controller's hard drive for more information.


14. Zero the Geometry Gap

Choose the zero gap icon , or select *Instrument>Gap>Zero Gap* and follow the directions on screen.

NOTE: The upper geometry should be at the testing temperature before zeroing the gap. This will account for the change in dimensions due to the coefficient of thermal expansion of the testing geometry/system.

If equipped with normal force, there are two options can be used to zero the gap, deceleration or normal force. These options are located in *Options>Instrument>Gap>Gap Zero Mode: Deceleration or Normal Force*. If using *Normal Force*, set the value equal to 1 N.

NOTE: If using Rheology Advantage 4.0 or greater and using the torsion rectangular system, move the clamps within 5 mm. Manually, align the fixed raised surface of each clamp and go to *Instrument>Bearing Lock* or

depress the Bearing lock icon . This will electronically inhibit any rotation of the geometry and allow one to zero the gap without the geometry rotating.

15. Gap Compensation

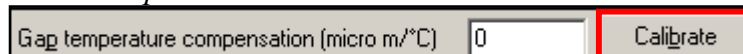
NOTE: Predetermined values can be entered by going to *Help>Index: 'compensation'*.

NOTE: The gap compensation check box must be activated, which is located within the *Options>Instrument>Temperature* dialog window, to account for the correction.

NOTE: Gap Compensation needs only to be used when testing over a temperature range.

NOTE: If controlling normal force throughout an experiment, the gap compensation value should be activated.

If using Rheology Advantage 3.0 and have the normal force transducer or 4.0 or greater with or without the normal force transducer, use the gap compensation button located in the *Geometry Page>Settings>Gap Temperature Compensation: Calibrate*.



If using Rheology Advantage 2.0 or lower and have the normal force transducer, perform an oscillatory temperature ramp procedure (Step 16). Using a torque of 0.1 μNm , a frequency of 1 Hz conducted at a ramp rate of 2°C/min under global normal force control (1N +/- 0.1, 1000 μm up/down, compression, set initial value) with a conditioning step temperature equal to the starting value of the temperature range of the experiment with an equilibration time equal to 5 minutes. Plot the data and then fit a straight line to the graph of Gap vs. Temperature. The slope must be then entered in the *Options>Instrument>Temperature: Temperature Calibration* region within

the cell located to the right of the appropriate temperature system.

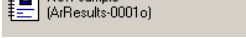
NOTE (version 2.0 or lower): The gap compensation value must be set to zero before this step is performed. Also, zero the normal force before running this gap compensation test.

16. Procedure

Create a new procedure by selecting *Procedure>New* or open a previously created procedure by selecting the appropriate file, by choosing *Procedure>Open*. The procedure can be viewed and adapted in the *Procedure*




17. Experimental Notes

Enter sample information within the *Notes Page*  or by selecting *Notes>New*.

18. Sample Loading

The amount of sample volume that is required, based on the dimensions entered in the *Geometry page>Dimensions* tab for cone, parallel and concentric cylinder systems, can be found in *Geometry page>Settings: Approximate sample volume*.

- When testing a dispersion or polymer melt when using the Peltier or Parallel Plate ETC, go to *Help>Video Clips: Loading and Trimming a Sample* or review **Steps 19 & 20**.
- When testing a polymer in pellet form, use the 25mm plates in conjunction with a melt ring, supplied in the Reusable Plate ETC kit, to contain the pellets. Place the melt ring around the lower 25mm stepped plate to form a well into which the pellets are placed, close the ETC, set a melt temperature , allow sufficient time for the pellets to melt, and then lower the gap appropriately, then follow **Step 19 & 20**.
- When testing a solid torsion sample using the ETC Torsion Kit: choose *Help>Content and Index>Index>Torsion>Guide to Sample Preparation*.

NOTE: Make sure that you run the compliance sample to determine the instrument compliance. This is determined by pressing the compliance calibrate button, located within the *Geometry Page>Settings>Compliance: Calibrate*.



This value should be less than 1.0E-2.

NOTE: Close the left ETC door and bend the upper moveable thermocouple (not the braided thermocouple attached to the heating elements) approximately 1.5 – 2 mm away from sample. Ensure that the thermocouple does not contact the geometry or the sample when the doors are fully closed.

- Concentric cylinder: Use the volume of material specified in the *Geometry page>Settings: Approximate Sample Volume*.
- Disposable ETC: Attach the plates loosely to the upper shaft and lower stage, in order for the plates to slide without slipping out of the holder, and then lower the plates to within 5mm. Activate normal force control



when viewing the *Instrument Status Page*, with settings: Normal force: 5N, Tolerance: 1N, Limit Up/Down: 100 μ m, Set initial value: On, Return to Window: On, Compression mode. Once the normal force has

reached 5N, then tighten screws, this will ensure that the plates are parallel. Then raise the upper geometry and load the sample following 18.a.

- f. Solid Sample Submersion System: Follow the Submersion Clamp Loading Wizard found in *Geometry page>Dimensions: Load sample*





19. Gap Closure



After loading a sample, the gap is closed by three different methods.

NOTE: When using the cone geometry, the gap set must be equal to the truncation gap value that is scribed on the geometry shaft.

NOTE: When using the parallel plate geometry, the gap is variable, and should be between 750 microns and Geometry Diameter (microns)/10.

- a. Manually enter the desired gap by selecting the enter gap icon  or by selecting *Instrument>Gap>Enter Gap...*
- b. Automatically have the instrument go to the gap value entered in the *Geometry Page>Dimensions* by using the go to gap icon  or select *Instrument>Gap>Go To Geometry Gap*.


NOTE: When using 19.a or 19.b, the compression settings (type, speed and distance above the geometry gap) can be adjusted by going to *Options>Instrument>Gap: Sample Compression*.

- c. Manually raise or lower the gap by using the   icons.

NOTE: These icons are only available when viewing the *Instrument Status Page*.

20. Trimming the Gap (cone or plate geometry systems)


Load extra material and close the gap to a value of 5% larger than the required gap, so that excess material is expelled from between the upper geometry, and lower the plate, i.e. overfilled state. Then hold the spindle knob with one hand, in order to keep the geometry from rotating, and trim the excess material using a right edged tool with the other hand. Then lower the gap to the final test gap, i.e. by 5% to allow the correct filled state to be achieved.

NOTE: If using Rheology Advantage 4.0 or greater, go to *Instrument>Bearing Lock* or depress the Bearing lock icon . This will electronically inhibit any rotation of the geometry and allow one to trim the sample without holding the spindle knob.



**Proper loading of sample after closing the gap for
cone and parallel plate geometry systems**

21. Run Test

Run test by selecting the run experiment icon  or choose *Experiment>Run*.

* To upgrade to the latest version of Rheology Advantage, please request the upgrade form from TA Instruments

Table 1.
Calibration
Guide

Instrument Inertia ^(Step 8)	Monthly Recommendation: Keep a log of this value
Geometry Inertia ^(Step 10)	Once during the geometry setup Verification of value is recommended daily, but not required
Bearing Friction Correction ^(Step 11)	Monthly Recommendation: Keep a log of this value
Rotational Mapping ^(Steps 13.a.i & 13.a.ii)	Once daily (if same geometry) Every time the geometry is changed
Oscillation Mapping [AR2000] ^(Step 13.b)	Only when using continuous strain control requiring low torque and displacement data
Zero Gap ^(Step 14)	Every time geometry is removed or replaced
Gap Compensation ^(Step 15)	Once if using same geometry/heating system

Support and Service

Website: www.tainst.com

TA Instruments Applications Support: 302-427-4070 (M-F 8:00-4:30pm EST)

TA Instruments Instrument Service Support: 302-427-4050 (M-F 8:00-4:30pm EST)