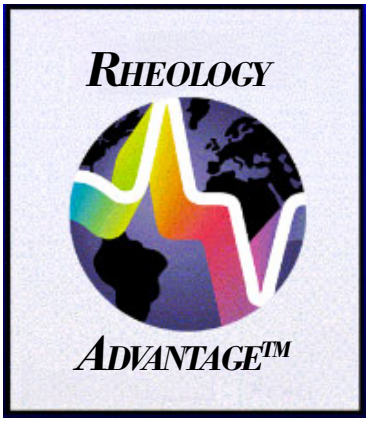
Rheology Advantage Instrument Control



Rheometrics Series Getting Started Guide

Revision E
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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 *Rheology Advantage*

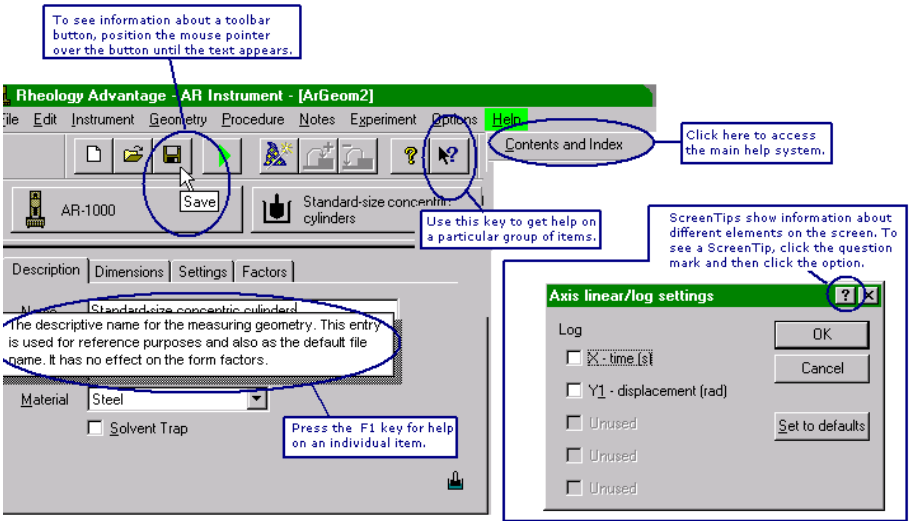
Welcome

Thank you for purchasing TA Instruments *Rheology Advantage*. *Rheology Advantage* makes it easy for you to set up and control the various instruments that are available from TA Instruments through the use of tool bars, menus, and windows. The Instrument Control program is used to run experiments on your rheometer and gather data files.

If you need help or information while using your *Rheology Advantage* software, online help is available as described in the next section.

Getting Help

Everything that you need to know about using the Advantage software is now online. This online information should be your primary source of information to help you use the programs. See the figure to the right for an graphical representation showing the location and types of help that are available.



Screen Tips

ScreenTips show information about different elements on the screen. To see a ScreenTip for a dialog box option, click the question mark and then click the option. (If the dialog box doesn't have a question mark button, select the option and then press the F1 key.) To see information about a tool bar button, position the mouse pointer over the button until the text appears (shown to the right).

Contents and Index

You can get Help using methods similar to the Windows® help system. Click **Contents and Index** on the Help menu. Click the **Contents** tab to scroll through a table of contents for the Help file. Click the **Index** tab to search for topics by using an index of subjects. Click on the **Find** tab to use full-text search and look for specific words or phrases.

Adding Annotations

You can add your own notes to the help system pages by using the **Notes** button. Any topic that has been annotated with notes will have a paper clip icon to the left of the topic title.

Navigating the Help System

Use the following icons to access more information when using the Rheology Advantage online help:

Provides a jump to more information.
Shows a popup with more information or an example.
Performs an action for you in the software.

Printing a Manual

During installation, you have the option to display one or both the icons for the TA Instrument Manuals and TA Software Manuals. These shortcuts allow you to access the library of available printable manuals for both hardware and software, respectively.

Changing the Help's Font Size

Follow these steps to change the font size for the current help file:

1. Open the desired help file. In an open Help topic window, click **Options**.
2. Select **Font** and then click **Small, Normal**, or **Large**.

Printing a Help Topic

Help topics can be printed by following these instructions:

To print one topic:

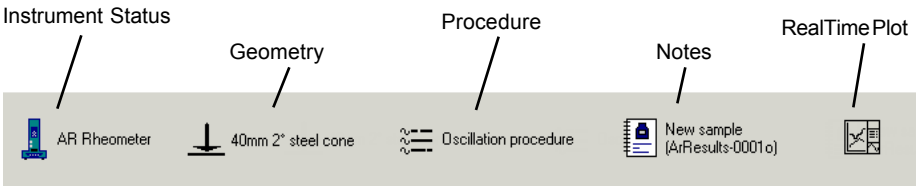
1. Open the desired help file and open a help topics. In the Help topic window, click **Options**.
2. Click **Print Topic**.

To print more than one topic:

1. Select **Help Topics**.
2. Select the **Contents** tab, then click on the book icon for the selected group of topics.
3. Click the **Print** button.

Overview

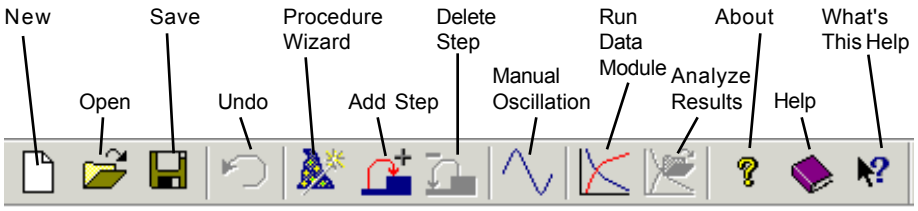
Five large buttons (tabs) control what is shown in the main display area of the Rheology Advantage Instrument Control program (shown below):



Button	Description
Instrument Status	Provides control over the basic instrument functions. This is a good place to manually assess a sample prior to procedure development.
Geometry	Shows the currently loaded measuring geometry and allows it to be edited.
Procedure	Shows the currently loaded procedure and allows it to be edited.
Notes	Provides sample information including density and optional user variables
Realtime Plot	If a measurement has been started, this displays the data graphically as well as numerical values for the last point.

Standard Tool Bar

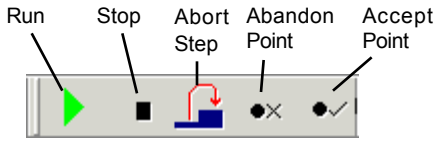
The tool bar (shown below) can be used for many of the same functions that are found in the menus. The table provides a list of the buttons available and a brief description of each one.



Tool Bar Buttons	Description
New, Open, Save	These buttons are multifunctional in that they can apply to the geometry, procedure, or notes depending on the active tab.
Undo	Click the Undo button to reverse the last command or delete the last entry and restores the previous text.
Procedure Wizard	Choose this button to start the wizard that will guide you through the process needed to set up a procedure.
Add and Delete Step	These buttons insert or delete the selected step(s) in the procedure. Changes can be made to the procedures even during the experiment as long as the step has not been started.
Manual Oscillation	Click this button to perform single point oscillation measurements. This is a good way to assess a sample prior to procedure development.
Run Data Module	Choose this button to switch to the data analysis program.
Analyze Results	Click this button to send the current data from the realtime plot to the data analysis program.
About	Select this to show the current program version and copyright information.
Help Topics	Click to access online help.
What's This Help	Position cursor on desired area, then click this button to access interactive help.

Experiment Run Tool Bar

The Experiment Run tool bar is used to control the experiment after setup. The buttons found on the tool bar are briefly described below.



Tool Bar Buttons	Description
Run	Starts the experiment.
Stop	Stops the experiment.
Abort Step	Terminates the current step and moves on to the next step in the procedure.
Abandon Point (Steady State Flow)	Skips the current point in the step without saving it to the data file.
Accept Point (Steady State Flow)	Overrides the steady state conditions in the step and accepts the point.

Configuring the Software

The Rheology Advantage software is configured with certain options in place when it is installed. This section tells you how to make various configuration changes to the software after installation.

Setting the Default Options

The following instructions provide information on setting up the desired default options to be used for the program:

1. Select **Options/Settings** from the menu.
2. Click on the **View** tab.
3. Click on the **Defaults** button.
4. Click the **Close** button.
5. Select **Experiment** from the menu.
6. Click on **Graph**, then select **Change variables...**
7. Click on the **set to defaults** button.

   NOTE: Steps 6 and 7 only apply to the active test type, *i.e.*, equilibrium flow etc. You must repeat this for each test type as required.

8. Click the OK button.

Viewing the Default File Locations

To view the directory that was last used for particular file types follow these directions:

1. Select **Options/Settings** from the menu.
2. Click on the **Directories** tab.

   NOTE: Directories can only be viewed using this method. To change directories you need to use the **Save As** function.

Using the Tool Bars

Tool bars give you quick access to various functions of the software. You can move them about the screen or "dock" them to the program screen. To find out the function of each button on a tool bar, hold the mouse cursor over the appropriate button until a pop-up text message appears.

Moving a Tool Bar

1. Click the move handle,  , on a docked tool bar or click the title bar on a floating tool bar .

2. Drag the tool bar to a new location. If you drag the tool bar to one edge of the program window, it becomes a docked tool bar.

Show/Hide a Tool Bar

1. Select **Options** from the menu.

2. Click on **General**.

3. Click on the **View** tab.

4. To show a particular tool bar, select (check) it on the list . To hide it, de-select it.

NOTE: To quickly hide a floating tool bar, click the close (x) button on the tool bar.

Changing the Button Size

1. Select **Options** from the menu.

2. Click on **General**.

3. Click on the **View** tab.

4. Select **Large Buttons** to increase the tool bar size.

Understanding the Menus

The following tables provide a description of the various menus used in the Rheology Advantage Instrument Control program.

File Menu

Refer to the following table for a brief description of the **File** menu:

Menu Item	Description
Open Session	Select this menu item to open a previously saved session. A session contains information on the geometry, procedure, notes, and instrument options. A session file can be used as a desktop short cut to automatically configure your rheometer on starting the software.
Save Session	This option saves the current geometry, procedure, notes, and instrument options in a single file.
Save Session As	This option also saves the current geometry, procedure, notes, and instrument options in a single file and it allows you to assign a new file name to the session file.
Load Options	Select Load Options from the menu to load the previously saved instrument options.
Save Options As	Use this function to save the current instrument options to a file that can be recalled at a later date.

NOTE: For information on menus not discussed here, refer to the remaining portion of this manual.

Instrument Menu

Refer to the following table for a brief description of the **Instrument** menu:

Menu Item	Description
Oscillatory Mapping	This is an advanced feature used to improve low torque/displacement oscillatory data. Refer to the document *New Features in Rheology Advantage 4.1* for more information.
Settings	This function provides information on the current instrument type and communications.

NOTE: For information on menus not discussed here, refer to the remaining portion of this manual.

Experiment Menu

Refer to the following table for a brief description of the **Experiment** menu:

Menu Item	Description
Graph	This function is used to change the properties, variables, and log/linear settings for the realtime plot. These functions perform in a fashion similar to the Rheology Advantage Data Analysis program. Please refer to the *Rheology Advantage Data Analysis Getting Started Guide* for further information.
Additional Variables	Select to access variables for the numerical data shown as part of the realtime plot window.
Previous Results	Used to display the last results file of the same procedure type or to select up to two control files.

NOTE: For information on menus not discussed here, refer to the remaining portion of this manual.

Options Menu

Refer to the following table for a brief description of the **Options** menu:

Menu Item	Description
Instrument Temperature	The AR 2000 automatically reads the currently installed temperature system. For other rheometers it is necessary to select the correct system using this function.
ID	Select this option to provide information on the system setup that may be requested by Service.
Experiment	Allows you to access settings that affect how data is stored and flagged as erroneous.
Settings	This function provides access to SI/cgs switch, range bars, language, and other global settings.

NOTE: For information on menus not discussed here, refer to the remaining portion of this manual.

Chapter 2
Preparing the Instrument

Before you can run a test on the rheometer, you will need to prepare the instrument for operation. Preparation involves proper calibration of the rheometer and selection of the appropriate geometry as directed in this chapter.

Calibrating the Rheometer

Your TA Instruments rheometer is a piece of precision scientific equipment and, as such, needs to be maintained to a sufficient standard so that operators of the instrument can be confident the data obtained from the instrument is correct.

A rheometer works with the following fundamental physical properties:

- Torque
- Displacement/Speed
- Temperature
- Gap
- Frequency
- Phase shift.

Since much of the rheometer calibration can only be adjusted using specialist tools, it is recommended that the instrument be given a full calibration/verification service at least annually. Although you can not perform a full calibration of the rheometer yourself, you can still monitor the performance and perform verification tests. It is recommended that you adopt some form of good laboratory practice (GLP) and establish a routine for monitoring the performance of the rheometer. The following areas should be considered:

- Verification of "System calibration factors."
  - — Instrument inertia.
  - — Measuring system inertia.
  - — Rotational (torque) mapping.
  - — Air bearing friction.
- Verification of Temperature calibration.

- Verification testing with a rheology standard (flow/creep/oscillation tests)

This chapter provides information that you will need to get started calibrating your rheometer. Refer to the online help available in the program for more details.

Calibrating Inertia

In an ideal world, whenever a torque was applied by a rheometer, it would act solely upon the loaded sample and nothing else. In practice, however, the non-zero moments of inertia of the rheometer spindle and measurement geometry mean that some of the applied torque is being used to accelerate or decelerate these mechanical components (until steady state is reached.)

A correction needs to be made to the torque value to reflect more accurately the conditions that the sample is undergoing. The amount of correction applied is based upon the calibrated values of the instrument and geometry inertia. In the oscillation mode, the correction is made automatically. In the flow mode, however, the correction can be toggled on or off as necessary. Flow inertia correction is more likely to be needed when low viscosity materials are being measured using fast ramps over a wide shear range.

Calibrating Instrument Inertia

Under perfect circumstances, the inertia value of the rheometer should not change with time. Running a calibration check on this value therefore gives a good indication of the performance of the rheometer. 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.

To calibrate the rheometer inertia, follow these steps:

1. Select **Options** from the menu.
2. Click on **Instrument...**
3. Click on the **Inertia** tab.
4. Ensure that there is no measuring system attached to the rheometer.
5. Click on the **Calibrate** button.
6. Follow the steps in the calibration wizard.

> NOTE: You can also type the value in manually at step 3, if required. (For example, you can type in a specific value to restore the calibration to a known calibration state.)

Measuring System Inertia

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

Follow these steps to calibrate the measuring system inertia:

1. Select **Geometry** from the menu.
2. Click on **View**.
3. Click on the **Settings** tab.
4. Ensure that the correct measuring system is attached to the rheometer.

5. Click on the **Calibrate** button (Geometry inertia).

NOTE: You can also type the value in manually at step 3, if required. (For example, you can type in a specific value to restore the calibration to a known calibration state.)

Performing Flow Inertia Correction

When a stress is applied to a sample it will move and continue to move while that stress is applied. If the stress is subsequently removed, the sample will stop moving, but not instantaneously. It requires a finite time to respond to this change in stress. This *lag time* is due to inertial effects and its magnitude is entirely sample dependent. In flow experiments, the effect of inertia is more pronounced in low viscosity fluids. Inertial effects manifest themselves in flow as the appearance of apparent thixotropic loops when a standard UP and DOWN flow curve is carried out. Consider the graph shown to the right.

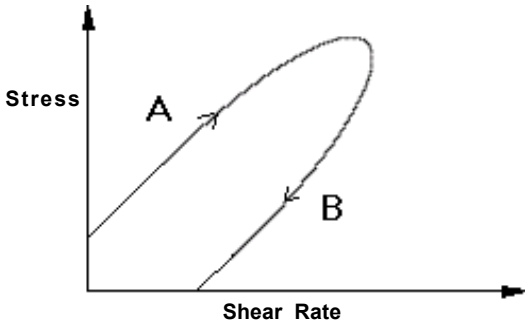


Figure 1
Flow Curve without Inertia Correction

You could interpret this graph as a flow curve on a thixotropic sample, highlighted by the presence of the hysteresis loop. However, the fact that the the DOWN curve (B) crosses the x axis alerts us to the fact that this apparent thixotropy is actually an inertial artifact and that the down curve intercepts the y-axis below the origin.

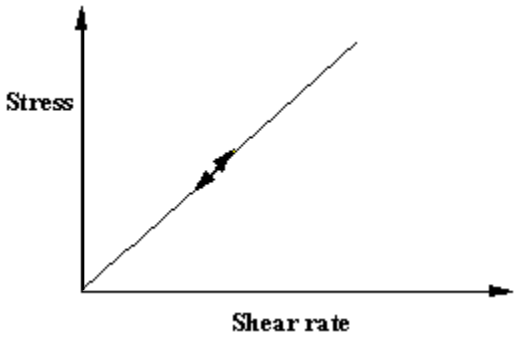


Figure 2
Flow Curve with Inertia Correction

The inertial correction feature available in the AR Rheometer eradicates such artifacts. It is a straight-forward toggle ON/OFF option that you can access using the software. (See the online help available for the software.)

Using the same sample as above, but measuring with the inertia correction ON, yields the results shown in Figure 2 to the left.

This now shows a Newtonian response, which is as expected. This inertia correction only needs to be active when low viscosity materials are being measured.

Using the Inertia Correction

The inertia correction is used automatically when carrying out oscillation tests. To use inertia correction in Flow mode:

1. Select **Options** from the menu.
2. Click on **Experiment**.
3. Select the **Flow** tab.
4. Ensure that both the Instrument and Geometry inertia have been calibrated.
5. Ensure that the **Inertia Correction** option is selected.
6. Press the **OK** button.

Rotational Mapping

Due to the micron tolerances needed to make an air bearing work, any bearing will have small variations in behavior around one 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.

To create a map, the program rotates the air bearing at a fixed speed, monitoring the torque required to maintain this speed through a full 360° of rotation. The number of points in the map (and the speed of rotation used) is dependent upon the mapping type used. These variations can then be allowed for automatically by the microprocessor, which is in effect carrying out a baseline correction of the torque. This results in a very wide operating range of the bearing without operator intervention—a confidence check in bearing performance. For maximum accuracy at the lower torque end of the rheometer, it is recommended that the mapping be performed each time a new measuring system is used.

To perform a rotational mapping:

1. Attach the required measuring geometry to the rheometer.
2. Load the correct Geometry file into the software.
3. Set the mapping type as follows:
    a. Select **Instrument** on the menu.
    b. Select **Rotational Mapping**.
    c. Select the **Bearing mapping** required [standard , fast, precision, or extended (AR 2000 only)—see "Mapping Types" on the next page for more information.]
    d. Select the number of iterations.
    e. Click **Perform Mapping** button—see "Mapping Types" on the next page for more information.

Mapping Types

The following mapping types are available in the Instrument Control program:

- **Standard mapping** takes approximately 1 minute to complete. It is suitable for day to day use.
- **Fast mapping** takes approximately 45 seconds to complete. Less mapping points per revolution are used than standard or precision mode. It is around half as accurate as Standard mode. It is suitable for day-to-day use where low-end accuracy is not so much of a problem.
- **Precision mapping** gives the most accurate calibration of the rheometer but takes around 2 minutes to complete. This mode is approximately twice as accurate as Standard mode.
- **Extended mapping** (AR 2000 Only) gives the best low torque/velocity performance.

Automatic Prompting for Mapping

The program can be set so that it will automatically prompt you to perform a mapping each time you change the installed measuring geometry. Follow these steps:

1. Select **Options** on the menu bar.
2. Select **Settings**.
3. Click on the **Mapping** tab.
4. Ensure that **Prompt user to map when geometry changed** option is selected.
5. Press the **OK** button.

Setting the Number of Mapping Iterations

For optimum performance at very low torques (<10 µN.m) multiple mappings can be performed. Generally, you will see little further improvement in performance after three consecutive mappings. To select the number of mapping iterations, use the following procedure:

1. Select **Options** on the menu.
2. Click on **Settings**.
3. Click on the **Mapping** tab.
4. Enter the number of mapping iterations required.
5. Click on the **OK** button.

NOTE: Extended mapping does not support multiple iterations.

Calibrating for Friction

An air bearing is used to provide virtually friction free application of torque to the sample. However, there will always be some residual friction. With most materials this friction 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 can be activated, if required.

Since each air bearing is custom designed and engineered, they are each unique. This means that the correction factor will also be unique to each bearing.

Determining the Friction Correction Factor

If you wish to apply the air bearing friction correction, you must first calibrate your bearing. This procedure should be carried out monthly if you use the option frequently. It can be affected by changes in the air supply so you are advised to ensure your air supply is stable. To determine the air bearing friction correction factor, use the following procedure:

1. Make sure the instrument inertia has been calibrated.
2. Attach the geometry of choice to the rheometer.
3. Enter the relevant geometry details via the software and make sure it has had its inertia calibrated.
4. Select **Options** from the menu.
5. Select **Instrument**.
6. Click on the **Miscellaneous** tab.
7. Click on the **Calibrate** button and follow the wizard.

Calibrating the Temperature

A full calibration of the temperature control system should only be carried out by an authorized TA Instruments representative. It is possible, however, to verify and adjust the calibration using a procedure such as given below.

1. Set the rheometer to give a value of 100°C.
2. Using a certified digital thermometer, measure the actual plate temperature.
3. Set the rheometer to 0°C.
4. Measure and note the actual temperature again. (Ensure that the temperature has stabilized.)
5. Repeat the process of setting and measuring temperatures, if more accuracy is required.
6. Determine the values for offset and span (see the next section).
7. Select **Options** from the menu.

8. Select **Instrument**.

9. Click on the **Miscellaneous** tab.

10. Click on the **Offset** entry for the selected temperature controller and enter the value obtained from the graph above for offset.

11. Click on the **Span** entry for the selected temperature controller and enter the span value obtained from the graph.

12. Click on the **OK** button to set these values on the rheometer.

Finding the Offset and Span Values

The temperature control devices on the rheometer can be independently calibrated by calculating a span and offset. It can be used in two ways.

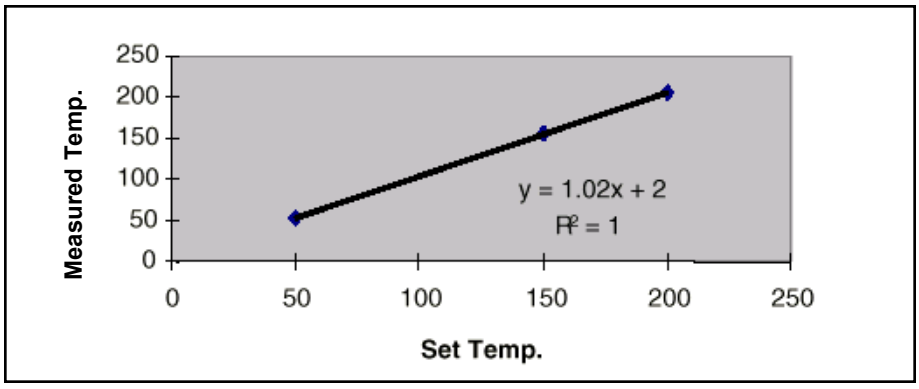
Offset & Span Calibration Method #1

Set a low and high temperature [ST1 & ST2] and measure the actual temperatures by another (reference) technique [RT1 & RT2]. If this is done at just two temperatures covering the temperature range of interest the calculation of span and offset can simply be done as follows:

Span = [ST2 - ST1]/[RT2- RT1]
Offset = ST2 - [RT2*Span]

If you want to make a more accurate calibration, you will need to measure at more than two temperatures. In this case, it is easier to use a spreadsheet (such as Microsoft® Excel.) Enter data into a table, then use the graph Wizard (or similar) to create an XY scatter graph. Insert the "best fit" line by right clicking on a data point. An example is shown below.

Set Temperature	Measured Temperature
50	53
150	155
200	206



From the equation of the best fit line [y = Slope*x + Intercept] calculate the span and offset as follows:

Span = 1/Slope = 1/1.02 = 0.98
Offset = -Intercept/Slope = 2/1.02 = -1.96

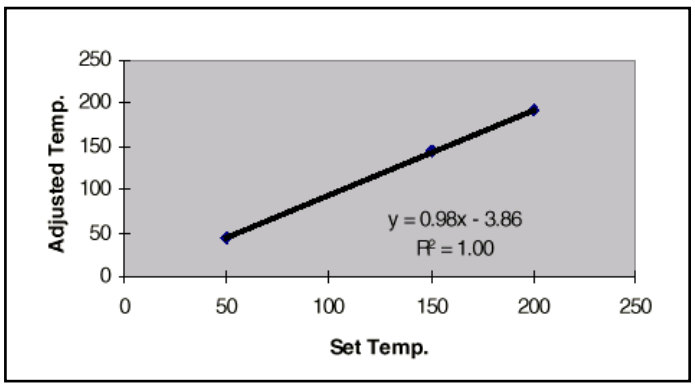
Offset & Span Calibration Method #2

The second way to calibrate the span and offset is to use a "standard" oil, which hopefully covers the temperature range of interest. Measuring viscosity at the set temperature doesn't help you. You need to adjust the set temperature until you get the certified viscosity value—some trial and error may be required. Note this temperature as the adjusted set temperature [AST]. For a two-point calibration calculate the span and offset as follows:

Span = [AST2- AST1]/[ST2-ST1]
Offset = AST2 - [ST2*Span]

If you have more than two points, use a spreadsheet as shown in this example.

Set Temperature	Adjusted Temperature
50	45
150	144
200	192



In this case there is no need for more calculation as:

Span = Slope = 0.98

Offset = Intercept = -3.86

System Verification

Although you cannot calibrate the rheometer yourself, you can check that the instrument is functioning properly by measuring the viscosity of a certified standard Newtonian oil. If you get a greater than 5% error in the reading, there is a possibility that your rheometer needs some attention from a TA Instruments Service Engineer. It is recommended that this verification check be performed at least on a monthly basis.

Carry out the following experiment.

1. Attach a 60 mm 2° cone to the rheometer. (This is the preferred geometry, if you do not have one, use the largest cone available to you.)

2. Install the measuring geometry into the rheometer.

3. Set the zero datum point for the measuring system.

4. Carefully load the sample, ensuring correct filling.

5. Carry out a "2-minute" flow test over as wide a range as possible.

6. Determine the Newtonian viscosity. If this value is more than 5% different from the certified value repeat the experiment. If the results are still in error, call your local TA Instruments office for advice.

The following are sources of error that can be checked:

- **Over or under filling of the gap**—ensure that you have established a standard method of sample loading.

- **Temperature error**—verify the temperature of the measuring geometry against a certified digital thermometer.

- **Gap setting**—ensure that the gap was correctly zeroed at the operating temperature.

Setting and Controlling the Gap

You may recall that the shear rate obtained in any experiment is due in part to the gap between the measuring systems used on the rheometer. Because of this, the accuracy of the way the gap is set is of critical importance to the accuracy of the results obtained. Whenever you change the measuring geometry on the rheometer, it is recommended that you ensure that the zero point or "datum" is correctly set. The rheometer can be made to obtain this setting automatically. Note that if the gap is incorrectly set, the data obtained will be in error. The actual gap that can be used depends upon the measuring geometry.

- For parallel plates, any gap (within reason) can be set.

- For cone and plates, the gap is determined by the cone truncation.

- Most other measuring systems also have a fixed gap setting.

When the test procedure calls for the temperature to change with time, the gap setting will change if the thermal expansion/contraction of the measuring system is not compensated for. The software, therefore, contains a thermal compensation mode that can be activated as required.

Thermal Compensation

When performing rheology testing, it is important to be able to compensate for changes in the temperature during the test measurement.

Why Use Thermal Compensation?

One of the features of the "Automatic Gap Setting" (AGS) facility is the ability to compensate for changes in the gap due to temperature changes during the measurement. The compensation is an active correction made to the gap—the rheometer physically moves the rheometer head to ensure that the actual gap remains constant.

The effects of expansion/contraction of the metallic parts of the rheometer can be eliminated by calibrating and then activating the thermal gap compensation factor in the software. For the most accurate compensation, you must obtain a value for each different measuring geometry and temperature module used.

Calibrating the Compensation Value

Follow the steps below to calibrate the compensation value:

1. Select and attach the required measuring geometry.
2. Click on the **Settings** tab of the Geometry template.
3. Click on the **Calibrate** button and follow the wizard.

Approximate Values

The table below gives an approximate guide to compensation values. These will generally give an acceptable performance for the gap compensation. It is only when you are working with extremely small gaps/cone angles and require the highest degree of precision, that you will need to calibrate these values.

Controller	Material	Value
Peltier	Stainless steel	0.5
Peltier	Aluminium	0.7
Peltier	Acrylic	0.5
ETM	----	0.9
ETC	Aluminum	2.9

Compliance

When measuring stiff materials, correcting for compliance can improve the results. For torsion measurements follow the **Calibration** wizard on the **Settings** tab of the Geometry template. For parallel plates, the default values are rheometer and measuring system-specific and it is recommended that you keep these values. The compliance correction is not available for cone and plate measuring geometries.

Finding the Zero (Datum) Point

Follow these steps to ensure that the zero point (datum) is correctly set:

1. Ensure that the air is applied and rheometer switched on.
2. Attach the required measuring geometry to the rheometer.
3. Load the Geometry file into the software.
4. Ensure that the instrument status page is active.
5. Select **Instrument** from the software menu.
6. Click on the **Gap** entry.
7. Select the **Zero gap** option or select the  button on the tool bar.
8. Position the measuring geometry within 5 mm of the lower plate. (Use the up and down arrows.)
9. Press the **Continue** button.

Setting the Geometry Gap

The actual gap used by a measuring geometry is not the same as the zero datum point, for instance a cone is backed away from the datum point by the truncation value of the cone.

1. Select **Geometry** from the menu.
2. Click on **View**.
3. Click on the **Dimensions** tab.
4. Enter the gap set value as follows:

   - Cones: Enter the truncation height (from the geometry certificate).
   - Plates: Enter the required gap.
   - Cylinders: Enter the gap (as specified on the geometry certificate).

Using the Backoff Distance Setting

When loading and removing samples from the rheometer, you must move the measuring geometry far enough apart to clean both the upper (rotor) and lower (stator) parts. The geometry file allows you to specify a *backoff distance*. This is the gap that the rheometer will go to when you press the backoff button  or select the **Raise head** option from the **Gap** section of the **Instrument** menu.

Set this value so that the gap is large enough to allow sufficient clearance for cleaning and gap loading. I t should not be so large as to cause a significant delay when the head moves between the backoff and geometry gap positions.

Adjusting the Rheometer Gap Velocity Values

The speed of movement of the rheometer head is controlled by the software. Three values can be adjusted.

Velocity	Description
Fine velocity	The velocity of the rheometer head when moving to the measuring geometry gap and less than 10 mm above the zero point. Above 10 mm, the Coarse velocity setting is used.
Course velocity	The velocity of the rheometer head when moving to gap and 10 mm above the zero point. Below 10 mm, the velocity slows to that selected as the Fine velocity.
Other velocity	The velocity of the rheometer head when manually closing the gap to within 5mm (for gap zero determination.)

To adjust these values select **Instrument** on the **Options** menu. Then click on the **Gap** tab.

Programmed Gap Closure

Under the tab for **Gap** on the **Instrument** item of the **Options** menu, you will see the settings for **Sample compression**. This setting allows you to decide how the gap is actually closed (either none, linear, exponential or normal force/stress). This is a very important consideration, particularly if you have thixotropic materials or samples with very delicate structure. Sometimes the mere action of loading the sample and closing the gap can destroy the very property you are trying to measure. See the table below for the available settings:

Sample Compression	Description
None	The gap is closed at a continuous velocity (as set under the Fine velocity entry.)
Linear	When the gap closes to the distance specified in the Compression distance entry, the gap is closed at the Compression velocity value.
Exponential	When the gap closes to the distance specified in the Compression distance entry, the gap is closed at an exponentially decreasing rate. The initial velocity is that specified in the Compression velocity entry.
Normal force/stress	Over the Compression distance, the velocity will slow at a rate proportional to the increase in the speed of normal force build up (based upon the entry peak normal force).

Solvent trap covers should be used if the sample is likely to dry out during a measurement or if solvent evaporation will occur. The relevant solvent is put into the solvent reservoir, once the cover is in place the free volume between the cover and the edge of the sample is saturated with vapor. An alternative is to "seal" the edge of the sample with silicone grease. However, you must ensure that the grease does not migrate into the sample.

Gap Setting Problems

If the rheometer is not performing a good gap set, consider the following points.

- Is dirt present on the measuring geometry? If so, clean it and try again.
- Is the Normal force reading out of range? Try zeroing the reading manually.
- Is the measuring geometry correctly located in the upper chuck? Try removing, cleaning the screw thread and then re-attaching it.

Chapter 3
Handling Files

This section provides you with information needed to open, create, and save information using the Rheology Advantage Instrument Control program.

What are the File Types?

The software uses the following file types:

Type	Description	Icon	Extension
Measuring geometry	Form factor information for the geometry.		*.argeom
Procedure, flow	An entire flow procedure, all steps.		*.arflow
Procedure, oscillation	An entire oscillation procedure, all steps.		*.arosc
Procedure,creep	An entire creep procedure, all steps.		*.arcreep
Notes	Sample notes.		*.arexpnotes
Results	Generated data.		*.rsl
Session	Saved sessions		*.ssar
Graph	Graph style settings		*.graphset
Options, Instrument	Instruments options		*.arinsopts
Options, Experiment	Experiment options		*.arexpopts
Instrument	Rheometer specific information.		*.arinstr

- **Measuring geometry files**: You must create a geometry file for each measuring system you have. You can also create separate files for the same geometry if, for instance, you wish to use different gap values or other settings.

(continued on next page)

- **Procedure files**: Procedure files contain the information required to run tests. Flow, creep and oscillation procedure files are distinguished by a different extension as well as using a different colored icon for each type.

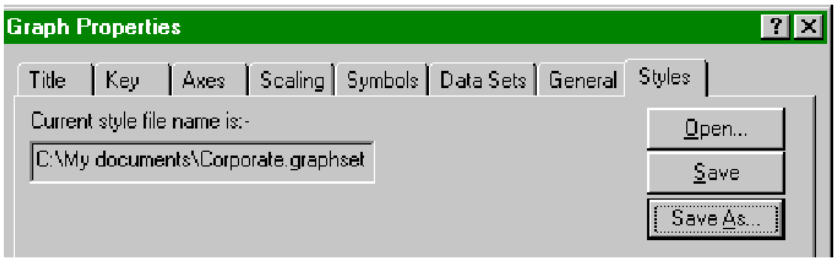
  Flow procedure files have the extension ARFLOW
  Creep procedure files have the extension ARCREEP
  Oscillation procedure files have the extension AROSC

- **Notes file**: All the information that you place in the Experiment notes section of the software is stored in a notes file.

- **Results file**: All data generated by the software can be saved as a results file. The file extension is the same, no matter what type of procedure was used to generate the data.

- **Session files**: Session files allow you to record all information active in the instrument software. A session file contains the following information:
  — Measuring geometry
  — Procedure
  — Notes
  — Instrument settings
  — Options.

- **Graph settings files**: The software allows you to create one or more "standardized" layouts for your graphs. These template files are stored as "Graph settings files" by use of the **Styles** tab (shown below) on the Graph properties.



- **Instrument files**: These files contain the serial port (comms) information required to connect to an instrument. A descriptive name is also stored in the file. Instrument files have the extension ARINSTR.

Choosing Filenames

To make it easier to find files, you can use long, descriptive filenames. The complete path to the file, including the drive letter, server name, folder path, and filename, can contain up to 255 characters. Filenames cannot include any of the following characters: forward slash (/), backslash (\), greater than sign (>), less than sign (<), asterisk (*), period (.), question mark (?), quotation mark ("), pipe symbol (|), colon (:), or semicolon (;).

Opening/Copying Files

You can open files from many different locations using the **Open** item under the **Geometry, Procedure,** and **Notes** menu items in the program. You can open a file on your computer hard disk or on a connected network drive. You can also open a file on a network, even if you don't have a connection to a network server, as long as your network supports UNC addresses.

1. Select **Open...** in the **Geometry, Procedure,** or **Notes** menu item.
2. Click the arrow next to the **Look in** box and locate the drive, folder, or Internet location that contains the desired file.
3. Click the file type you want in the **Files of type** box.
4. Double-click any folders until you open the folder that contains the file you want.
5. Double-click the file you want to open or select the file and click **Open**.

Opening a File Using a UNC Address

1. Select **Open...** in the **Geometry, Procedure,** or **Notes** menu item.
2. Click the file type you want in the **Files of type** box.
3. Type the UNC address for the network share containing the file that you want in the **File name** box. Press ENTER. For example, type \\Computer1\Files to open a file on the server Computer1, on the share Files.
4. Double-click any folders until you open the folder that contains the file you want.
5. Double-click the file you want to open or select the file and click **Open**.

Opening a Rheology Solutions File

1. Select **Open...** in the **Geometry, Procedure,** or **Notes** menu item.
2. Click the arrow next to the **Look in** box and locate the drive, folder, or Internet location that contains the desired file.
3. Select the Version 1 option in the **Files of type** box.
4. Double-click any folders until you open the folder that contains the file you want.
5. Double-click the file you that want to open or select the file and click **Open**.

Creating a Backup Copy of a File

1. Select **Open...** in the **Geometry, Procedure,** or **Notes** menu item (depending on what you wish to back up.)
2. Select **File/Save As...**
3. Choose a location and type in a name for the backup file.
4. Click the **Save** button.

Using the File Property Information

File properties are details about a file that help identify it—for example, a descriptive title, the author name, the subject, and keywords that identify topics or other important information in the file. You can set file properties for any Rheology Advantage file. You can also create custom file properties relevant to your own products or information handling routines.

The following are the types of file properties:

- **Pre-set file properties:** These are properties such as author, title, and subject that already exist for you to enter a value.
- **Custom file properties:** You can create custom file properties that include any value you assign. For example you could create a custom file property to use in all QC Verification files.
- **Automatically updated file properties:** Statistics such as file size and the dates files are created and last modified are file properties that are automatically maintained for you. These file properties help you search for all files created after a certain date, such as March 3, 1996, or last modified yesterday.

Viewing the Property Information

To view the file property information for an already *loaded file*, follow these steps:

1. Click on **Geometry, Procedure,** or **Notes** on the menu.
2. Select **Summary** information.

To view the file property information for a *stored file*, follow these steps:

1. Select **File/Open...** from the menu or click the  button.
2. Click the arrow next to the **Look in** box and locate the drive, folder, or Internet location that contains the desired file.
3. Select, then right click the file you want to view file properties for.
4. Select **Properties** from the pop-up menu.
5. Click on the tabs to see specific information.

Adding/Editing the File Properties

To add or edit the file property information for an already *loaded file*, follow these steps:

1. Click on **Geometry, Procedure,** or **Notes** on the menu.
2. Select **Summary information**.
3. Add or change the properties on the **Summary** tab.

To add or edit the file property information for a *stored file*, follow these steps:

1. Select **File/Open...** from the menu or click the  button.
2. Click the arrow next to the **Look in** box and locate the drive, folder, or Internet location that contains the desired file.
3. Select, then right click the file you want to set file properties for.
4. Select **Properties** from the pop-up menu.
5. Add or change the properties by selecting the options you want on the **Summary** and **Custom** tabs.

Creating Custom File Properties

1. Select **File/Open...** from the menu or click the  button.
2. Click the arrow next to the **Look in** box and locate the drive, folder, or Internet location that contains the desired file.
3. Select, then right click the file you want to set file properties for.
4. Select **Properties** from the pop-up menu.
5. Click the **Custom** tab.
6. Type a name for the custom property in the **Name** box or select a name from the list.
7. Click the type of property you want in the **Type** box.
8. Type a value for the property in the **Value** box. The value you enter must match the selection in the **Type** box. For example, if you click **Number** in the **Type** box, you must type a number in the **Value** box. Values that don't match the property type are stored as text.
9. Click the **Add** button.
10. Click the **OK** button.

Chapter 4
Operating the Instrument

This chapter provides the information needed to operate your rheometer using the Rheology Advantage Instrument Control program. In order to run the instrument you will need to design a test, set up and operate the rheometer and make sure that you have achieved accurate temperature control. In addition to the information already described, this chapter also provides you with the instructions needed to remove the air bearing clamp, level the rheometer, and manually operate the instrument.

Designing a Test

When using the rheometer to analyze test samples, the most common method used to obtain results is by creating or editing procedures. This section deals with the concepts of test design.

What Steps are Involved?

A procedure consists of one or more steps that are executed in sequence. Each step can be made to conditionally end, if required. The following is a description of the types of steps that can be used in a procedure.

- **Conditioning step**: Use this step to ensure that the sample is at the correct temperature and any residual normal force (from loading) is at an acceptable level. You can then "pre-condition" the sample. This allows you to apply a shear to remove any history associated with the sample preparation, storage and loading. Additional conditioning steps can be included in your procedure.

- **Main procedure steps**: In between the (optional) conditioning and post-experiment steps, you can define one or more main procedure steps, which can be of any type and in any order.

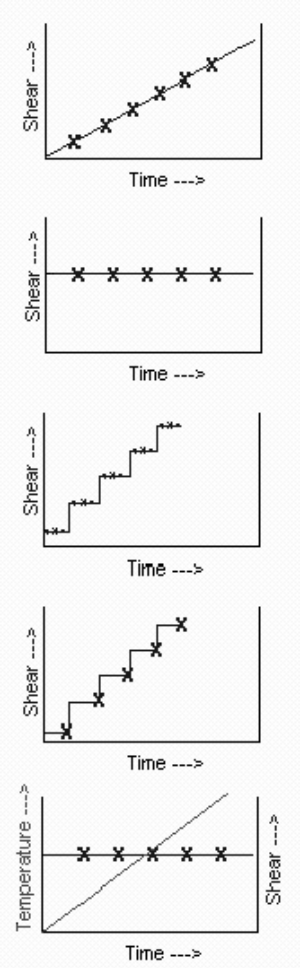
- **Conditional step termination**: The program allows you to define conditions in which a step is halted ahead of its normal termination conditions. You can use this to ensure that, for instance, the instrument does not over speed or apply excessive strains.

  Rather than running a step for a certain amount of time, you may wish to run it until stable data is obtained. You can set an equilibrium limit (such as the viscosity value becoming constant when running a single shear with time) that will stop the currently active test.

- **Post-experiment step**: You can set the temperature system to idle. You can also get the rheometer to head towards a new temperature. This can be handy as an aid to cleaning the rheometer.

Available Flow Steps

The following steps are available in the Test type option for flow mode:



Continuous
Continually ramps the shear
and samples at defined intervals.

Peak hold
Holds the shear constant and
samples at defined intervals.

Steady state flow
Applies successive shear values.
Data sampled under equilibrium conditions.

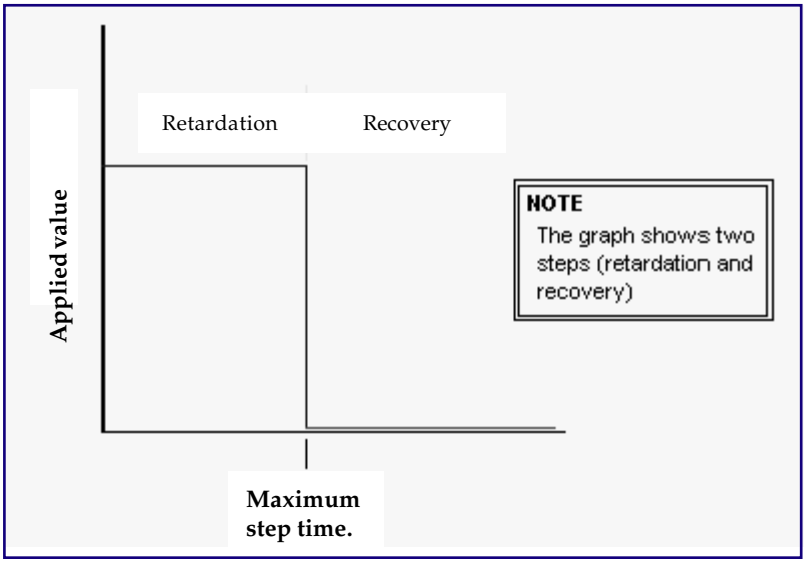
Stepped flow
Applies successive shear values.
Data sampled at end of each value.

Temperature ramp
Holds the shear constant
while rampling the temperature.
Samples at defined intervals.

If your instrument has a normal force transducer, the squeeze/pull off test will also be available. In this test the upper geometry is either raised or lowered at a constant speed and the force is measured.

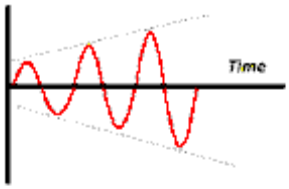
Available Creep Steps

The creep test function is shown to the right.

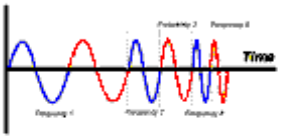


Available Oscillation Steps

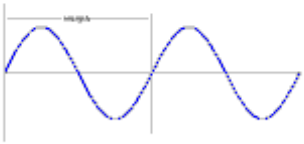
The following steps are available in the **Test type** option for oscillation mode:



Strain/Stress Sweep
Applies a fixed frequency and increments the amplitude.



Frequency Sweep
Applies a fixed amplitude and changes the frequency.

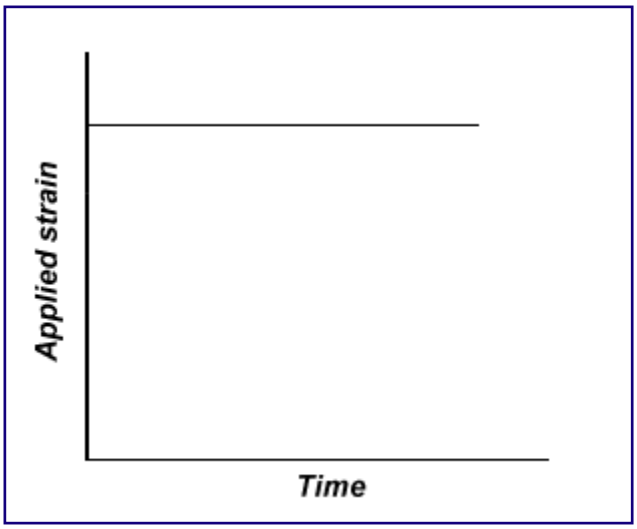


Temperature Ramp/Time Sweep
Applies a fixed frequency with a set amplitude. Monitors viscoelastic properties as a function of time/temperature.

Time/Temperature Sweep
As per ramp, but applies a sequence of discrete temperature steps rather than a smooth ramp.

Available Stress Relaxation Steps (AR 2000 Only)

The stress relaxation or step strain test is shown to the right.



Manually Operating the Rheometer

When the Instrument Control program displays the **Instrument Status** page you can manually set and control the following aspects of the rheometer. You can access the parameters to set from the **Instrument** menu item by clicking on the appropriate icon or by clicking on the grid.

- Set Temperature
- Set Stress/Torque
- Set Rate/Speed
- Set Strain (AR 2000 Only)
- Zero Strain
- Zero Normal Force.

There are a number of gap controls that can also be accessed from the **Instrument Status** page:

- Raise head (to back off distance)
- Go to geometry gap
- Enter gap
- Zero gap
- Stop head
- Up and down.

Rotational mapping, normal force control, and bearing lock/release (AR 200 only) can also be accessed from the **Status** page.

Display Raw (Point) Data During a Test

Use the following steps to display the point graph for Oscillatory and steady state flow procedures.

Displaying Data for a Flow Procedure

1. Select **Options** from the menu.
2. Click on **Experiment**.
3. Click on the **Flow** tab.
4. Select the option **Display point graph during run**.
5. Click on the **OK** button.

Displaying Data for an Oscillation Procedure

1. Select **Options** from the menu.
2. Click on **Experiment**.
3. Click on the **Oscillation** tab.

4. Select the option **Display waveform during run**.

5. Click on the **OK** button.

The point graphs can also be saved to disk and be viewed on a point-by-point basis in the data analysis program.

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