

**RSA-G2
Solid Analyzer:
TA Instrument Help (partial)**

November 17, 2023

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1 Environmental control

Setting the Temperature

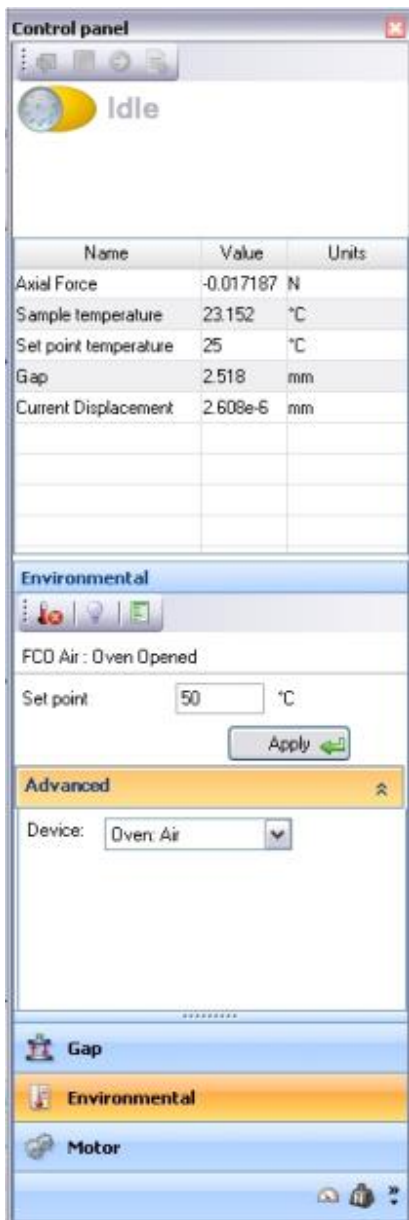
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Although the experiment temperature is defined in the test procedure, it is often desirable to set the temperature and other **environmental** control options prior to starting the experiments. These parameters can be changed by accessing either the **Environmental** tab of the **Control panel** within TRIOS (screen shown below) or the RSA-G2 Temperature Control panel on the touch screen. The options in these panel vary based on **environmental** option installed.

Within these windows you can perform the following operations:

- Monitor current status
- Change temperature set point
- Enable/disable **Environmental** Control Unit
- Change gas source options
- Enable/disable FCO
- Fill LN2
- Enable rapid cooling for LN2
- Turn on/off FCO oven light



See Also

[Introducing the RSA-G2 Environmental Control Options.](#)

Changing the Temperature Set Point

It may be desirable to alter the temperature set point outside of a test condition; for example, one may wish to preheat the geometries at the experimental temperature prior to zeroing the gap. This can be accomplished by changing the temperature set point on either the TRIOS **Control panel** or the RSA-G2 Temperature Control touch screen window.

To change the temperature set point using the TRIOS **Control panel**:


1. [Open](#) the TRIOS **Environmental** Control panel.
2. Enter the desired value in the **Set point** field, then click **Apply**.

To change the temperature set point using the ARES Temperature Control touch screen window:

1. [Open](#) the Temperature Control touch screen window.
2. Click **Set Temp** to update the set point value.
3. Click **Exit** to close the window.

Enabling/Disabling the Environmental Control

To enable or disable the **environmental** control units (for example, FCO) using the **Environmental** Control panel:

1. [Open](#) the TRIOS **Environmental** Control panel.
2. Click **Enable/Disable environmental control** .

To enable or disable the **environmental** control using the RSA-G2 Temperature Control touch screen window:

1. [Open](#) the Temperature Control touch screen window.
 2. Click **Temp Enable** to toggle the enable/disable state.
- NOTE: When the **environmental** control unit is enabled, the **Temp Enable** light is green. A yellow light indicates that the device is disabled.

Some notes on enabling or inhibiting **environmental** control using the RSA-G2 keypad **Environment** button:

- Press the **Environment** button on the keypad to enable or inhibit **environmental** control.
- The indicator adjacent to the keypad button lights **RED** when environment control is inhibited, or **GREEN** when enabled.
- The **Environment** keypad button provides a hardware-based inhibit of all environment heating and cooling operations. If the **RED** Environment indicator is lighted, then **environmental** heating and cooling are unconditionally inhibited, and cannot be enabled under software control from TRIOS or from the LCD interface.
- When inhibited, the only way to re-enable **environmental** control is to press the **Environment** button on the keypad, which removes the hardware inhibit and changes the indicator color to green. If the LED does not switch to green, then the instrument has detected a safety issue that must be corrected before the environment system can be enabled (such as over-temperature, loss of temperature control, invalid device identification, etc.). An environment-inhibit condition cannot be cleared remotely in order to protect the safety of the operator and the instrument.
- If any temperature control device is plugged in to one of the temperature option connectors on the instrument during power-up, then the instrument will start in the environment-inhibited state. This is a safety measure to prevent unexpected temperature operation during an unattended power-up.

Switching the Gas Source

FCO heaters and LN2 Controller heaters operate using gases during tests. See below for information on changing the gas source for these accessories.

To Switch the FCO Gas Source

The FCO heaters use either gaseous nitrogen (N2) or air during a test.

- When using the optional LN2 control, you can also specify LN2. Selecting LN2 supplies evaporated LN2 from the LN2 Controller.
- When using the optional Chiller Panel, you can also specify Chiller.

To switch available gas source options from the TRIOS **Environmental** Control panel:

1. [Open](#) the TRIOS **Environmental** Control panel.
2. Click the **Advanced** drop-down arrow.
3. Select the gas source from the **Device** drop-down list.

To switch available gas source options using the Temperature Control touch screen window:

1. [Open](#) the ARES Temperature Control touch screen window.
2. The yellow indicator appears next to the current gas source selection. To change the selection, click the desired option. Options will vary based on installed accessory.
 - LN2 operation: choose between **Off, Air, Gas, LN2**.
 - Chiller operation: choose between **Off, Air, Gas, Chiller**.
3. Click **Exit** to close the window.

To Switch the LN2 Controller Gas Source

When using the optional LN2 Controller you can select to flow gases through the heaters in either of the following two ways:

- Use evaporated liquid nitrogen (LN2) at all times
- Use liquid nitrogen until a specified temperature is reached, then use either nitrogen gas or air (depending upon which gas input port is selected with the Gas Supply to Oven Selector Switch).
- NOTE: When using the gas switching option, ensure that the input temperature for changing from LN2 to gas is approximately 10 degrees higher than the temperature for changing from gas to LN2. This prevents the system from switching back and forth between the two sources at the input temperature, as switching from liquid nitrogen to gas causes a momentary change in temperature.

To enable the gas switching option, follow the instructions below:

1. [Open](#) the TRIOS **Environmental** Control panel.
2. Click the **Advanced** drop-down menu.
3. Select the desired gas source from the **Device** drop-down menu. Choose between: **LN2 Only, LN2-Gas Source, or LN2 - Air Source**.
4. If you wish to switch between gas sources both in upward (heating) and downward (cooling) direction, select Options from the Environment Control panel to access the **ARES G2 > Main** options screen.

Filling the LN2 Accessory

To initiate a filling of the LN2 Accessory with coolant using the **Environmental** Control panel:

1. [Open](#) the TRIOS **Environmental** Control panel.
2. Click the **Advanced** drop-down menu.
3. Click the arrow to the right of the **environmental** control unit status, and select **LN2 Fill**.

To initiate a filling of the LN2 Accessory with coolant using the Temperature Control touch screen window:

1. [Open](#) the ARES Temperature Control touch screen window.
- NOTE: If the **LN2 Fill** indicator light is yellow, the LN2 Cooler requires filling. If the **LN2 Fill** indicator light is green, enough coolant remains in the LN2 Cooler.

2. Click **LN2 Fill** to fill the LN2 Cooler, if necessary.
3. Click **Exit** to close the window.

Enabling LN2 Rapid Cooling

When the LN2 Rapid Cooling option is enabled, the LN2 Cooler evaporates liquid nitrogen at the maximum possible rate.

- NOTE: **LN2 Rapid Cooling** is always on at temperatures lower than -124°C.
1. [Open](#) the TRIOS **Environmental** Control panel.
 2. Click the **Advanced** drop-down menu.
 3. Click the arrow to the right of the **environmental** control unit status, and select **LN2 Rapid Cooling**.

Turning On/Off FCO Oven Light

To turn on/off the FCO Oven light:

1. [Open](#) the TRIOS **Environmental** Control panel.
2. Click **Turn Oven Light On/Off** .

To turn on/off the FCO Oven light using the Temperature Control touch screen window:


1. [Open](#) the ARES Temperature Control touch screen window.
2. Select the **Environmental** tab, and press **Oven Light** to toggle turning the oven light on and off.

Using the Environmental Control Panel

The TRIOS **Environmental** Control panel (shown below) provides real time temperature parameters and control operations related to this component. The functions of the controls related to the **environmental** control unit in use. The Environmental Control panel is divided into the status section and control section, as shown below.





Follow these instructions to use access this panel:

1. Select **Experiment** tab > **Controls**  > **Environmental** tab on the **Control panel**.
2. Use the controls on the panel to perform various functions as described below.

RSA-G2 Environmental Control Panel Functions

Item	Function
Enable/Disable environmental control	Turns on or off temperature control.
Turn Oven Light On/Off	Toggles turning the oven light on/off.
Options	Shortcut to Environmental Options.

<p>Set temperature</p> 	<p>Enter a value and click Apply (the green arrow).</p>
<p>Advanced</p> 	<p>FCO: Select the desired gas source.</p> <p>LN2: Select the desired gas source; enable filling and rapid cooling.</p>

Using the Touch Screen Temperature Control Panel

The touch screen **Temperature Control** panel provides the ability to view the real time temperature parameters as well as control operations related to this component. The functions of the controls related to the **environmental** control unit in use. Follow these instructions to access this window:

1. Select the **Instrument** tab on the [RSA-G2 instrument touch screen](#).
2. Touch the **Set Temp** button. The screen shown in the figure to the right is displayed.
3. Use the controls on the panel to perform various functions as described below.

This panel provides the following functions (refer to the callouts below):

1. View current temperature setpoint
2. Change the temperature setpoint
3. Monitor and change the current gas source.
4. Monitor the fill level of the LN2 and initiate a fill.
5. Monitor and change the FCO state (enable/disable)



2 Gap control

Setting the Sample Gap

In this topic

[Zeroing the Gap](#)

[Manually Zeroing the Gap](#)

[Automatically Zeroing the Gap](#)

[Setting the Gap](#)

[Using the TRIOS Gap Control Panel](#)

[Using the Touch Screen Gap Control Panel](#)

Zeroing the gap between upper and lower test geometries (except for cone geometries) is a prerequisite to setting the gap during sample loading. Zeroing the sample gap requires lowering the stage until the upper and lower geometries touch, then setting the gap value to read zero millimeters. After that, moving the stage results in an accurate gap indication as displayed by the gap value. Contact is determined by looking for a change in the axial force value. The sample gap should be re-zeroed any time a new geometry is mounted or the test temperature is changed. The RSA-G2 can take account for changes due to geometry and sample expansion with temperature during tests, but always begins with the assumption that the sample gap is correct at the start of an experiment. For example, if running a test at 200°C, the geometry should be heated and sufficiently equilibrated at that temperature to ensure the thermal expansion of the tool is accounted for when establishing a zero point. Failure to accurately zero the sample gap can have an adverse effect on the accuracy of the calculated data in certain test geometries.

Set the gap by either using [TRIOS software](#) or the [RSA-G2 touch screen](#). This topic details with both methods. The procedures described below are general for all geometries. Specific details regarding each geometry are described under that geometry's individual section.

Zeroing the Gap

Two methods are available for zeroing and setting the gap: [Manual Zero](#) and [Auto Zero](#). For most applications, the Auto Zero method is recommended.

If testing is to be done at temperatures other than ambient, install the geometry and then adjust the temperature to the desired initial value. Allow the geometries to reach thermal equilibrium before zeroing and setting the gap. For best results, the fixtures should be equilibrated at the approximate gap to be used during testing.


Manually Zeroing the Gap

To manually zero and set the gap between upper and lower geometries, perform the following steps:

1. Ensure that the upper and lower geometries are clean and properly mounted.
2. Tare the transducer using the [Transducer Control panel](#).
3. Open the **Gap**Control panel from either [TRIOS](#) or from the [RSA-G2 Instrument Touch Screen](#) options.
4. Using the RSA-G2 keypad, lower the stage using the slew (rapid) mode until the distance between the upper and lower tools is about 0.5 mm, as determined visually.
5. Using the step (slow) mode of the Gap Control, lower the stage until the normal force **just begins** to deflect from zero, indicating that the tools are in contact with one another.
6. Access the TRIOS [Gap Control panel](#). Click **Tare gap** to offset the current gap value to zero.
7. Raise the stage to a height that allows the sample to be loaded. The sample can now be loaded. When the stage is lowered, the correct distance between geometries is displayed.

Automatically Zeroing the Gap

Establish a zero reference gap between upper and lower geometries as follows:

1. Ensure that the upper and lower geometries are clean and properly mounted.
2. Open the **Gap**Control panel from either [TRIOS](#) or from the [RSA Instrument Touch Screen](#) options.
3. Using the RSA-G2 keypad, lower the stage using the slew (rapid) mode until the distance between the upper and lower tools is about 0.5 mm, as determined visually.
4. Select **Zero Fixture** .
 - When the gap zero routine is initiated, the instrument tares the normal force, closes the geometries until they touch with 1000 grams force, calibrate linear system compliance (in the normal or z-axis), makes additional measurements to establish zero position of geometry, and backs off the upper geometry to the specified gap position set from the TRIOS [RSA G2 Options](#).
6. Raise the stage to a height that allows the sample to be loaded. The sample can now be loaded. When the stage is lowered, the correct distance between geometries is displayed.

Setting the Gap

After zeroing the gap, set the gap to a specific value using the procedure below. To manually move the stage, open the **Gap Control** panel from either [TRIOS software](#) or from the [RSA-G2 Instrument Touch Screen](#) options.

1. Raise the stage and place the sample onto the lower tool.
2. Open the **Gap** Control panel from either [TRIOS](#) or from the [RSA Instrument Touch Screen](#) options.
3. Enter the following information:
 - a. **Set gap**: Enter the desired gap position. The maximum allowed Axial Force used during gap setting operation can be changed in the TRIOS [Options](#) > [RSA G2 Options](#) window.
 - b. **Maximum force**: The option to set this parameter is only available from the TRIOS [RSA G2 Options](#) window (click the **Options** icon in the TRIOS gap panel to access). Enter the maximum allowed Axial Force to use during the gap setting operation. When the absolute value of the Axial Force exceeds this value, the stage motion stops until the magnitude of the Axial Force drops below this value. Stage movement then resumes.

When setting the gap, the "Maximum Force" option provides two benefits. First, it allows an operator to set the gap in a repeatable way. Loading the sample the same way each time leads to more reproducible results between different operators, or with different geometries. Second, it helps avoid internal pre-stressing a sample during loading, which again, could lead to erratic or inconsistent results. This feature is also helpful when loading soft samples that may squeeze out from between the test tool if too much force is applied.


4. Select **Go to Geometry Gap**. The stage descends quickly until the upper tool is close to the Set Gap Position. At this point the rate of descent slows until the gap is achieved. The gap is set when the Gap Status returns to **Idle**.

As the upper assembly lowers and the sample is compressed, the maximum force limit will be maintained as the sample relaxes (if the maximum force limit is met before the set gap is reached). Should the set gap not meet within a reasonable time, the experiment can be started with the measured gap at the maximum force. Alternatively, if the gap must be set, increase the maximum force and/or reduce the sample size.

Using the TRIOS Gap Control Panel

The **Gap** Control panel (shown below) provides the ability to control stage and gap operations. The Gap Control panel is divided into a status section and control section, as shown below.

Follow these instructions to access and use this function:

1. Select the **Experiment** tab > **Controls** , then select **Gap**.
2. Use the **Gap** Control panel to perform the various functions described below.

RSA-G2 Gap Control Panel Functions

Item	Function
Stop Head	Stops any head movement.
Go To Geometry Gap	Moves the head to the value specified in the geometry gap\truncation gap field for the current geometry.
Go to Loading Gap	Moves the head to the value specified in the loading gap field for the current geometry.
Zero Fixture	Runs the zero gap routine.
Tare Gap	Tares the current gap position to zero.
Tare Transducer	Used to tare both the torque and normal force transducers. Generally used in conjunction with Axial force adjustment or gap setting functions. On the RSA-G2 touch screen, yellow indicator lights appear next to Tare Torque and Tare Normal on the Instrument tab when an offset is in effect. Note: this control does not bias the fast torque and normal force signal during the data acquisition process.
Remove Transducer Tare	Removes the transducer tare.
Head up	Moves head up while button remains pushed.
Head down	Moves head down while button remains pushed.
Options	Shortcut to Gap Options.
Set gap	Enter a value and click Apply (the green arrow).

See Also

[Axial Force Guidelines](#)

Using the Touch Screen Gap Control Panel

The **Gap Control Panel** on the RSA-G2 touch screen also provides the ability to view the real time gap and normal force parameters as well as control operations related to these components, such as Set Gap and Zero Fixture.

Observe the current gap value and status display above **Go To Gap**. If you wish to adjust the sample gap using the touch screen panel, follow these instructions:



1. Select the **Instrument** tab on the [RSA-G2 instrument touch screen](#).
2. Touch the **Gap Control** button. The screen shown in the figure to the right is displayed.
3. Use the controls on the panel to perform various functions as described below.

Touch Screen Gap Control Panel

Item	Function
Gap	Enter the desired gap value in mm using the numeric buttons below the entry field. Rcl = recall Clr = clear
Go To Gap	Select this button to move the RSA-G2 stage to the specified gap. See Also: Setting the Gap
Zero Fixture	Use the Zero Fixture button to establish a zero reference gap between the upper and lower fixtures. This function is generally performed before the start of each test with clean fixtures, before the sample is loaded. See Also: Zeroing the Gap
Stop	Use the Stop button to stop the motion of the stage.
Exit	Use the Exit button to return to the main touch screen.



3 Axial force guidelines

Axial Force Adjustment Guidelines

In this topic

[Introduction](#)

[Axial Force Control and Temperature](#)

[Gap Compensation](#)

[Controlling Axial Force](#)

Introduction

Axial force control is an important feature for many applications, from ensuring that contact is maintained between the **geometry** and sample during compression control to keeping solids taut in tension control. This topic describes how axial force control operates, explains the axial force control parameters, and cites specific conditions to apply when testing materials requiring axial force control.

When Should Axial Force be Used?

Axial force control should be used when materials expand (and/or contract) or slip during testing. If changes in volume occur, the material will either be subject to a normal stress field or shrink away from the testing **geometry**; either situation will cause errors in the results. Generally, volume changes within a material occur by changes in temperature, solvent/moisture loss, or when the material gels or cures. In order to convey the use of axial force completely, the remainder of this section will associate the volume change of a material with a change in temperature, described by the coefficient of expansion. The most widely used test mode to control axial force is in the oscillation mode with a temperature ramp. Axial force control is also used to maintain a static force on the sample to avoid unloading of the sample during oscillation testing.

- NOTE: Axial force control is not applicable when using cone and plate type **geometry**, since correct results depend on the truncation gap to remain constant when using this **geometry**.

How Does Axial Force Control Work?

The axial compliance of the ARES-G2, RSA-G2, DHR, and AR is only a few tenth of microns per Newton. The normal force transducer senses volumetric changes of the sample. A volumetric change entraining a vertical expansion of a few microns, assuming the material is stiff enough to translate the resultant force, may cause the normal force transducer to go into an overload condition. If axial force control is active, the normal force transducer will detect the change in normal force and command an adjustment of the head position i.e. increase or decrease the gap to hold the actual normal force constant.

- NOTE: In case of the ARES-G2 and RSA-G2, the axial force control is disabled by default during measurement in order to provide unbiased data. Axial normal force is only applied prior to and/or after the data sampling cycle. In order to enable continuous axial force control, check the box **Maintain axial force during acquisition** in the Advanced section of the test form. In the case of the DHR and AR Rheometers, the axial force is continuously adjusted; only in the DMA mode (axial displacement) the axial force control is disabled during the oscillation measurement. The axial (static) force reported in this case is the force prior to the oscillation measurement..

Axial Force Control and Temperature Gap Compensation

ARES-G2 and DHR behave differently during axial force control when the thermal compensation coefficient (Gap Hold) in the **geometry** form is checked. On the ARES-G2, the Gap Hold function is deactivated as soon as the axial force control is activated. On the DHR, the thermal gap compensation (Gap Hold) remains active when axial force control is active as long as the axial force stays within the lower and upper band of the window defined by the current axial force and the force sensitivity.

- NOTE: When the thermal gap compensation coefficient in the **geometry** form is a non-zero value and the box is not checked: on the ARES-G2, Gap Hold is deactivated, but the reported gap is numerically adjusted for the thermal expansion; on the DHR, the thermal gap compensation is disabled completely. In order to achieve this condition on the ARES-G2, the gap compensation coefficient must also be set to zero.

Controlling Axial Force

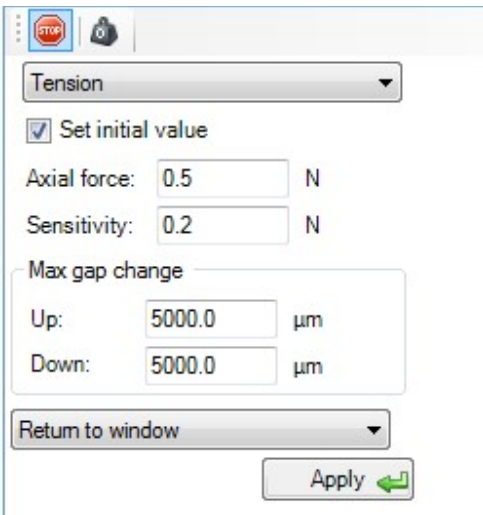
Axial force control can be enabled/disabled and set in the **Axial force** section of the **Instrument** Control panel (maintaining the specified conditions prior to the test), as well as in a procedure. Axial force adjustment conditions set in the **Instrument** Control panel can be inherited and used during the test procedure, if desired.

Instrument Control Panel Settings

Axial force control prior to the start of a test procedure is set in the **Axial force** section of the Control panel. Activate axial force control after loading the sample, but before enabling temperature control. Select the

Experiment tab > **Controls** , then select **Axial Force**.

Axial force control settings for RSA-G2, ARES-G2, and DHR for linear and rotational oscillation testing:



The screenshot shows the 'Axial Force' control panel. At the top, there is a 'Tension' dropdown menu. Below it is a checked checkbox labeled 'Set initial value'. The 'Axial force' is set to 0.5 N, and the 'Sensitivity' is set to 0.2 N. Under the 'Max gap change' section, both 'Up' and 'Down' are set to 5000.0 μm. At the bottom, there is a 'Return to window' dropdown menu and an 'Apply' button with a green arrow icon.

The axial force control panel only allows you to set a constant force. Advanced axial force control algorithms that may be needed during testing must be set in a procedure conditioning step.

- NOTE: When the box for **Set initial value** is not checked, the axial force will only be enabled when the measured axial force is outside of the window defined by the axial force \pm the sensitivity. As such, the sensitivity settings can be used to delay the start of axial force control until the measurement force exceeds the window.
- Prior to the test (when no test procedure is active), the axial force is controlled continuously.

Procedure-Based Control

In order to control axial force during a procedure, the axial force control must be enabled in a procedure. If axial force adjustment is not activated in the procedure conditioning step, axial force control is disabled at the start of the procedure. Insert a **Conditioning** > **Options** step and select the **Axial Force Adjustment** option in order to activate this feature. When axial force is not active prior to the test start, there is no need to add a conditioning step to disable the axial force control.

Control Panel vs. Procedure Settings

For many testing scenarios, axial force needs to be active prior to and during a procedure. In this case, the axial force needs to be activated and set in the Control panel and in a procedure conditioning step. The adjustment conditions prior to the start of the test can be inherited by the procedure. Different control conditions can be set if desired in the procedure. Use **Inherit** for the force adjustment option only if you want to keep the same constant force during the complete test procedure. If a different force, or advanced axial force control algorithms need to be set, switch the axial force adjustment to **Active** and set the desired control parameter.

Turning Off Axial Force Control When the Test is Running

The axial force parameters inherited from the Control panel or set in a procedure conditioning step can be turned off during the procedure by adding a **Conditioning** step and selecting **Disabled** for the Axial force mode.

Axial Force Control Settings in the Procedure Step

- Axial force control settings for DHR standard mode (rotation oscillation testing):

1: Conditioning Options

Axial force adjustment

Mode

Tension Compression

Axial force N Set initial value

Sensitivity N

Advanced

Gap change limit up μm

Gap change limit down μm

Return to window Return to initial value

Purge gas only (no active cooling)

- NOTE: On the DHR (standard mode), the axial force control is always active (not interrupted during data acquisition) during a test procedure.
- Axial force control settings for ARES-G2 standard mode (rotation oscillation testing):

2: Conditioning Options

Axial force adjustment	
Mode	Active ▾
<input checked="" type="radio"/> Tension	<input type="radio"/> Compression
Axial force	1.0 N <input checked="" type="checkbox"/> Set initial value
Sensitivity	0.1 N
	<input type="checkbox"/> Compensate for stiffness changes
Advanced	
Max gap change up	3.0 mm
Max gap change down	3.0 mm
<input type="radio"/> Return to window	<input checked="" type="radio"/> Return to initial value
Priority	<input checked="" type="radio"/> Data sampling <input type="radio"/> Force control
Adjustment time out	5.0 s

Auto strain adjustment	
Mode	Disabled ▾

- NOTE: On the ARES-G2, the axial force control is only adjusted prior to the next dynamic point. During the actual data acquisition, the axial force is not adjusted. This can be overrun by checking the box **Maintain axial force during acquisition** on the Advanced tab of the test mode procedure step. In order to avoid disturbance of the torque signal in the acquisition phase, we recommend that you not use this feature. Check **Compensation for sample stiffness** to change the static axial force based on the estimated shear modulus of the new data point. The algorithm extrapolates the value of the static force based on the modulus change and a scaling factor. This is useful for torsion measurements during the glass transition to avoid unnecessary stretching of the sample. The **Scaling factor** sets the strength of the static axial force dependence as a function of the modulus change. If the scaling factor is zero, the axial force remains constant. If the scaling factor is one, the axial force changes proportional with the modulus. Usually the static force is desired to change less rapidly than the modulus. A good value of the scaling factor is 0.6. This provides a moderate change of the static axial force with the change in modulus.
- Axial force control settings for RSA-G2, ARES-G2, and DMA mode (linear oscillation testing):

1: Conditioning Options

Axial force adjustment	
Mode	Active ▾
<input checked="" type="radio"/> Tension	<input type="radio"/> Compression
Axial force	0 N <input checked="" type="checkbox"/> Set initial value
Sensitivity	0 N
Proportional force Mode	Force Tracking ▾ <input type="checkbox"/> Compensate for modulus
Axial Force > Dynamic Force	1.0 %
Minimum axial force	1.0 N
Programmed Extension Below	0 Pa
⌵ Advanced	
Max gap change up	10.0 mm
Max gap change down	1.0 mm
<input checked="" type="radio"/> Return to window	<input type="radio"/> Return to initial value
Priority	<input type="radio"/> Data sampling <input checked="" type="radio"/> Force control

In DMA mode, the axial force and the dynamic force are measured by the same normal force sensor. Therefore, the axial force must be adjusted prior to the measurement of the individual dynamic point. In order to avoid too much stretching or compression of the sample, the axial force is adjusted proportional to the dynamic force (force tracking mode). When **Compensate for modulus** is selected, the static force is based on the estimated modulus rather than the dynamic force. This is necessary when **Auto strain adjust** is activated and the strain changes during the test. The algorithm extrapolates the value of the modulus at the new temperature based on the previous modulus change. The new dynamic force is calculated from the estimated modulus for the applied strain. The new static axial force is then determined from the new dynamic force based on the **Axial Force > Dynamic Force** criterion.

The **Programmed Extension Below**, when used, applies when the measured modulus falls below a threshold value. When the modulus crosses the threshold value, the axial force control is disabled/enabled because the sample has become too soft/stiff for the axial force control operation. The sample is extended (to avoid buckling or squeezing) by changing the gap based on the material expansion coefficient (set in the **Options > Material properties**). If this value is zero, the extension is based on the applied gap change before the **Programmed Extension Below** criterion came into effect.

- Axial force control settings for DHR DMA mode (linear oscillation testing):

1: Conditioning Options

Axial force adjustment

Mode	Active ▾
<input checked="" type="radio"/> Tension	<input type="radio"/> Compression
Axial force	5.0 N <input checked="" type="checkbox"/> Set initial value
Sensitivity	0.1 N
Proportional force Mode	Force Tracking ▾
Axial Force > Dynamic Force	25.0 %
Minimum axial force	0.5 N

Advanced

Gap change limit up	5000.0 μm
Gap change limit down	5000.0 μm
Adjustment time out	5.0 s

Purge gas only (no active cooling)

The axial force control on the DHR is based on the same algorithms as the ARES-G2/RSA-G2. The instrument always operates in **Data sampling** priority mode with an **Adjustment time out** for the axial force adjustment control.

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4 Conditioning steps

Setting Up Conditioning Steps

In this topic

[Overview](#)
[Transducer](#)
[Stress Control](#)
[Sample](#)
[Options](#)
[End of Test](#)

Overview

Conditioning tests are passive test types in the sense that no test is performed or no test data is generated. Conditioning tests are usually added as the first step in a procedure and can also be inserted among tests to change settings. There is no need to insert a Conditioning test unless the current default conditions should be overwritten. The Condition step controls instrument (motor position, transducer zero acquisition) and environmental (temperature) conditions, as well as test performance conditions such as axial force, strain adjustment (auto strain), stress control tests (creep and recovery), and delays (equilibrium time).

The following settings can be selected in the conditioning step:

- [Transducer](#): Transducer step controls transducer mode and zero acquisition.
- [Stress Control](#): Stress Control step is used to determine or load motor control constants for the Stress Control tests.
- [Sample](#): Sample step enables delays and environmental settings.
- [Options](#): The options step enables axial force adjustment and auto strain adjustment settings.
- [Dielectric](#): The DETA step is used to DETA simultaneously with mechanical a test.
- [End of Test](#): Use this step to change instrument environmental, transducer, motor, and axial force conditions at the end of the procedure.

Transducer

From the transducer control panel, the transducer can be configured for operation when no experiment is running, such as when loading the sample and adjusting the temperature. For testing, the transducer is automatically configured according to the test type executed. Unless there is a reason to override the default setting, there is no need to use the transducer conditioning block setup.

If you want to change the transducer settings during a test or save the transducer settings in a named procedure, insert the transducer conditioning block.

The transducer conditioning step is divided into 3 sections, which can be activated or deactivated individually by checking the box.

1. **Transducer Mode Switching**: Check the box to activate the Transducer Mode Switching. If unchecked (and the transducer conditioning block is the first block in a procedure), the default transducer setting are used: the mode from Control panel is inherited. (See [RSA-G2 Default Transducer Settings for Procedures](#) for additional information.)

Configuration: When the Transducer Switching Mode is selected, the following two transducer settings are possible:

- **Maintain:** If this mode is selected, the transducer mode maintains the setting from the Control panel, or from the previous step.
 - **Override:** If this mode is selected, the setting are changed to either **Spring** (soft) or **FRT** (stiff).
2. **Motor State / Equilibration Delay:** Check the box to activate the motor state and equilibrium function. Note that at this moment, the motor must be set in the zero position, otherwise the test cannot be started properly.
- **Equilibration time:** Activate the equilibration time by entering a value in the **Equilibration time**.
3. **Transducer Zeroing / Range Switching:** Check the box to activate this transducer range selection and zero acquisition. If unchecked, the **High** range with zero acquisition is used. When the transducer switching mode is selected, two settings are possible: **Maintain** or **Override**.
- **Maintain:** Maintains the setting from Control panel or previous step without zero acquisition.
 - **Override:** specify your desired settings.
 - **Range selection:** Select the transducer range from **High** (0.2 to 3500 g), **Low** (0.05 to 350 g), or **Auto-range**. **Auto-range** only works for oscillation tests, and is not applicable for other tests like transient and axial tests. Due to the air turbulence in the oven, the low end of force for both **High** and **Low** range does not have much difference (around 0.2 g); thus, the **High** range is always recommended for use with the FCO. The default setting is **High** range.
 - **Transducer zero time:** The zero time is the time required to establish the zero signal of the transducer by averaging a signal over a period of time and setting it to zero. By default, this step is performed at the beginning of every test procedure with **Fast** time (about 2 seconds). **Standard** time is about 7 seconds and **Precision** is about 20 seconds. Longer zero time will give you a more accurate zero signal. In case you wish to override the default settings, you can insert a **Conditioning > Transducer** as the first step in a procedure. For oscillation tests, an accurate force zero is not essential because only the peak-to-peak force is important. In this case, the default option (**Fast**) is recommended. For transient tests (like step strain and step stress), an accurate zero is important, and the **Precision** time may be needed.
 - **Zero force:** Select this check box if you want to zero force prior to starting the experiment and if you have changed other transducer settings; otherwise, zero acquisition will not be performed (skipped).

Stress Control

The Stress Control block sets the control conditions for Stress Control led tests such as the creep and stress ramp test modes. This block must be inserted into the procedure prior to any Stress Controlled test. Use the Stress Control conditioning block to:

- Run an oscillation pre-test, and calculate and download the control parameters to the instrument.
- Load the precomputed control constants and download these to the instrument. Use this selection when the pretest has already been performed and the control constants have been saved.
- NOTE: When running a stress control test, establishing a correct force condition of the sample is very important. For most cases, the separation of the pre-test is more convenient and easy to set up.

Sample

The Sample conditioning step is used to condition the sample. Sample conditioning includes temperature equilibrium, delays for sample stabilization, or preshearing.

Environmental Control Option

Select the following environmental control parameters:

1. **Temperature:** Enter the desired temperature value, or select **Inherit value** to maintain the previously specified temperature at the start of this step.
2. **Wait for temperature:** Select this option to wait until the entered temperature is reached before beginning data collection. If you wish to begin data collection while still not achieving the temperature, disable (uncheck) this option.
3. **Soak time:** This value is the amount of time to delay data collection at the start of the step, typically to allow for temperature equilibration and/or sample relaxation. If **Wait for temperature** is not selected, the time is measured from the start of the step. If **Wait for temperature** is selected, the time is measured from the point at which the temperature reaches the commanded temperature. Because of the mass of the sample, geometries, and environmental control systems, a “soak time” (i.e., time to equilibrate at temperature) is recommended, particularly when starting experiments at non-ambient temperatures or when the temperature is changed significantly between steps. When the axial force control (auto-tension) is active, the **Soak time** also serves as the adjust time for the first data point. This time is also used at each increment temperature.

Equilibration options

Check the **Equilibration** box to activate this function. Specify an equilibration time.

Options

The Option conditioning step is used to set the axial force and the auto strain adjustment. These functions are only active in oscillation tests and at the first soak time period for other tests.

[Axial Force Adjustment](#) allows the control of the axial force during a test. The axial force can be controlled in either tension or compression. It is essential for axial force control to provide a proper static force to keep the sample from buckling (or slipping from contact with the fixture) and over-stretching during the testing. The static force must be larger than the oscillation force for a meaningful oscillation measurement. RSA-G2 provides constant force and force tracking modes for an accurate axial force control.

[Auto strain adjustment](#) changes the strain amplitude in order to hold the measured force within the operation range of the transducer or the force range specified by user. Auto strain adjusts the strain only within the specified strain range when the oscillation force is out of the specified range.

Axial Force Adjustment

1. **Mode:** Select one of the three choices. Depending on the option selected, other parameters may need to be specified.
 - **Disabled:** Axial force control is disabled in subsequent steps.
 - **Active:** Axial force control is enabled using the specific force value as the commanded axial force to maintain during subsequent steps.
 - **Inherit:** Use this when you desire for the current axial force and other parameters applied to the sample to be inherited based on the last setting (either from the Control panel or previous **Conditioning > Options** step).
2. Select between: **Tension** and **Compression:** Define the direction of axial force control by selecting the appropriate radio button. For example, select **Tension** for tensile fixtures, and **Compression** for compression parallel plates or bending fixtures.
3. **Axial force** and **Set initial value:** Enter the value for the axial force. Check **Set initial value** if you want the axial force control to apply the entered value immediately; without checking **Set initial value**, the axial force control will be delayed to start once the axial force reaches the entered value.

4. **Sensitivity**: The sensitivity defines the axial force window for adjustment. The Axial force adjustment is active only when the measured force falls outside of this window.
5. **Programmed extension**: When the measured complex modulus drops below a critical value (user-defined), the axial force control turns off and a Gap Temperature Compensation mode switches on. In this case, the stage moves at an instrument-measured thermal expansion coefficient (average 3 data points around threshold) to compensate for the thermal expansion if the zero value of the material thermal expansion coefficient is entered; otherwise, the stage moves at the total thermal expansion of geometries and materials entered by the user. Note that once **Programmed extension** is triggered, stays activated until the end of the test, even in the event that the modulus comes back over the threshold value.
6. Use the **Advanced settings** for further refinement of the axial force control.
 - **Max gap change up and down**: The maximum gap change upwards and downwards defines the active displacement window available to the axial force adjustment algorithm. As soon as the gap falls outside of this window, the axial force adjustment is deactivated independent of the value of the axial force.
 - **Return to window or command force** : When the axial force control becomes active, the algorithm can be set to continue adjusting the force until the force is within the sensitivity window or until the set axial force is reached. Select the appropriate radio button.
7. **Priority**: Sets the priority to **Data sampling** or axial **Force control**.
 - **Data sampling**: If selected, the data sampling algorithm overrides the axial force control. After a user defined **Adjustment time out**, data sampling is resumed, whether the axial force adjustment is completed or not. Use this option if a continuous and timely sampling of oscillation data points is desired. Note that the measured axial force may be outside of the desired sensitivity window.
 - **Force control**: If selected, data sampling will wait until the axial force control algorithm is finished. Use this option if accurate control of the axial force prior to the data sampling is desired. Note that data points might not be sampled in a continuous fashion, so gaps in time for the stream of data points, usually at transitions, may occur.

Note that in general cases, **Data sampling** with 5 seconds adjust time is highly recommended.

Auto Strain Adjustment

1. **Mode**: Select one of the two choices:
 - **Disabled**: Auto strain adjustment is switched off. Use this setting when you do not want to use this feature.
 - **Enabled**: AutoStrain adjustment will be used in the subsequent Oscillation test modes. Enter the desired parameters below.
 2. **Strain adjust**: This is the amount of strain added or subtracted from the actual strain amplitude for each adjustment step. The amount is given in percent of the last strain applied.
 3. **Minimum strain**: This defines the lowest strain amplitude to be allowed on the sample.
 4. **Maximum strain**: This value defines the maximum strain amplitude to be allowed on the sample.
 5. **Minimum torque**: This field defines the minimum torque amplitude to be allowed on the sample.
 6. **Maximum torque**: This field defines the maximum torque amplitude to be allowed on the sample.
- NOTE: The strain limit overrides the force limit. This means that the maximum strain will remain the upper strain limit, even when the force falls below the minimum force.

End of Test (EOT)

The End of Test (EOT) conditioning block is the last step in a procedure. It is used to define the conditions of the environmental system, transducer, motor and stage control after the procedure is completed.

- **NOTE:** The end of test condition step is a passive step and sets the conditions when the test ends, either normally, user terminated, or instrument terminated. If you want the end of test conditions applied even if the test is terminated prematurely, the EOT conditioning block should be placed at the beginning of the procedure. If it is desired to change the EOT conditions based on when the procedure is terminated, more than one EOT can be used in a procedure. The position within the procedure in reference to the step when the procedure is terminated defines the EOT conditions that are applied.

Environmental Control

Choose one of the following selections for the Temperature Control:

- **Maintain:** The environmental system remains at temperature, no change
- **Disable temperature control:** The environmental system is switched off
- **Go to temperature:** A control field to enter the new temperature is displayed

Select following for the FCO Gas Source (only valid if the FCO is the installed environmental system):

- **No change:** Do not change the FCO gas source
- **Disable FCO:** Disable the FCO environmental system
- **Enable air:** Enable air source for the FCO
- **Enable gas:** Enable the gas source for the FCO
- **Enable LN₂:** Enable the liquid nitrogen controller for the FCO
- **LN₂/air switch:** Switch gas source from the liquid nitrogen controller to the air supply
- **LN₂/gas switch:** Switch gas source from the liquid nitrogen controller to the gas supply
- **Maintain LN₂ fill:** This selection is always available. Check **Enabled** if you want the LN₂ controller Dewar to remain ready for immediate operation

Axial force adjustment

Select the following options when checked:

- **Disable:** Disable the active axial force control
- **Active:** Activate axial force control after the procedure is terminated. Make the following selections:
 - Choose **Tension** or **Compression** for the direction of the axial force control
 - Set the **Axial force** for axial force control

Transducer/Motor

Override the default settings as follows if checked:

- **Transducer range:** Maintain or select either **High range** or **Low range**
- **Transducer Normal Force Mode:** Maintain or select **FRT** or **Spring**
- **Motor lock:** Maintain or select **Locked** or **Unlocked**



5 Test: Axial

Setting Up an Axial Test

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Overview

Axial tests deform the sample, while controlling the vertical motion of the slide. The dynamics of the axial tests depend on the performance of the motor for the slide motion. Either the axial (normal) force or the vertical displacement is controlled.

- In the “constant force/stress” mode, the extension or the **compression** of a sample is measured upon the application of a constant positive (compression) or negative (tensile) force.
- In the “linear displacement” mode, the sample is compressed or extended at constant clamp speed (mm/s) and the force required is measured.
- In the “exponential displacement” mode, the sample is compressed or extended at exponentially increasing (decreasing) speed (Hencky rate).

Typical applications for the axial tests are the tack test, lubricated squeeze flow on polymers, strain/stress curves on films and fibers, etc.

In a tack test a constant force is combined with a linear rate test. In the first part the sample is compressed at constant force then the plates are separated at constant speed. The force as a function of displacement is measured. Maximum force and adhesion energies can be determined from this test procedure.

The lubricated squeeze test uses the exponential **compression** mode. In order to eliminate shear contributions at the plates, a lubricant is put between the sample and the plate. From the force a biaxial extensional viscosity can be calculated. In the squeeze or rim shear experiment, the shear deformation between the plates (compression) at the edge of the sample is held constant.

Test Setup

To select an Axial test, select **Other - Axial** from the **Procedure** tab option. See [Using Experimental Procedures](#) for detailed instructions.

When you perform an Axial test, the following parameters need to be chosen.

Environmental Control

Select the following environmental control parameters:

1. **Temperature**: The temperature selection to maintain throughout the test. Enter the desired test temperature, or select **Inherit Value** to maintain the previously specified temperature at the start of this step. The temperature range is dependent on the configuration of your instrument and the installed environment control system.
2. **Soak time**: The amount of time to delay data acquisition at the start of the step, typically to allow for temperature equilibration. This time is measured from the start of the step if **Wait for temperature** is not selected, or from the point at which the measured temperature becomes stabilized at the specified **Temperature**, if **Wait for temperature** is selected. Because of the mass of the sample, test fixtures,

and environmental control systems a "soak time" (*i.e.*, time to equilibrate at temperature) is recommended, particularly when starting steps at different temperatures. A five-minute soak time is sufficient for most samples in cases where the change in temperature is not too large. This time is also used at each increment temperature.

3. **Wait for temperature:** Select this option to wait until the measured sample temperature reaches the specified temperature before beginning data acquisition. If you wish to begin data collection without waiting for the sample temperature to reach the specified value, disable this option.

Test Parameters

Set up the following test parameters:

1. Enter the **Duration** time for your experiment.
2. Select the Motor direction (**Tension** or **Compression**).
3. Select from the following list:
 - **Constant force:** Enter the desired force (positive values indicate tension and negative values indicate **compression**) that will be a constant value maintained throughout the test. The value should exceed the maximum dynamic force expended during a test.
 - **Constant stress:** Enter the desired steady shear stress (positive values indicate tension and negative values indicate **compression**) that will be maintained for the specified time period.
 - **Constant linear rate:** Enter the desired linear rate (positive values indicate tension and negative values indicate **compression**) that will be maintained for the specified time period.
 - **Hencky strain rate:** Enter the desired strain rate (positive values indicate tension and negative values indicate **compression**) that will be used for the specified time period. This parameter applies a constant rate of sample strain. Linear displacement rate is adjusted to maintain a constant sample rate. Applies deformation at a constant strain rate, in units of reciprocal seconds as defined by the following equation:

$$\dot{\epsilon}_h = \left(\frac{1}{L} \right) \left(\frac{dL}{dt} \right)$$

$$\dot{\epsilon}_h = \text{Hencky Strain (1/s)}$$

$$L = \text{sample length (mm)}$$

4. **Maximum gap change:** This value is the maximum allowable gap range that the head will move to maintain the set force value.
5. Choose between **Log or Linear** sampling:
 - **Logarithmic:** The logarithmic method uses the entered value to spaced logarithmically. Logarithmic sweeps can be run in ascending or descending order. Enter the desired number of **Sample points**. The number of points determines the total number of data points collected.
 - **Linear:** The linear method uses the entered values as the starting and ending range of the test, with intermediate points calculated by adding or subtracting the increment until the final value is reached. Select the desired method used to collect your data.
 - ◆ **Sampling rate:** This method is preferred when you plan on collecting larger amounts of data. Enter the desired number of data points collected per second.
 - ◆ **Sampling interval:** This method is preferred when using low rates of sample collection. Enter the desired number of seconds between each data point.
 - ◆ **Sample points:** Enter the total number of sample points you wish to collect over the entire test time.

Data Acquisition

Save image: Select to store images of the test within the data file for future recall when using the Camera accessory.

- **Save image every:** Specify the collection rate for time-based experiments. Select the minimum time in seconds between images. An image is stored with the next test point when the time between images has elapsed.

Step Termination

TRIOS Software allows you to define conditions in which a step is halted ahead of its normal termination conditions (Limit checking). 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.

Test mode steps can be conditionally terminated by checking the limit or the equilibrium of test variables. The procedure will continue with the next procedure step. When a test mode is conditionally terminated, an event log is created with the termination details.

1. Check the box to enable **Limit checking** termination. Select the test variable from the list, the condition (< smaller than or > larger than) and the value.
2. Check the box to enable the **Equilibrium** termination. Select the variable to track for equilibrium, the tolerance in % and how many data points in sequence have to fulfill the criterion before the test step is terminated.

- NOTES:

- When both **Limit checking** and **Equilibrium** conditions are checked, the test is terminated by the condition that comes first.
- In a Multiwave test the test termination uses the fundamental value of the test variables only (not the harmonic values).
- The test termination process is not synchronized with test mode. Since a test mode only stops after completion of the current data point acquisition cycle, additional data points can still be collected after the test termination condition has been fulfilled.



6 Test: Frequency sweep

Setting Up an Oscillation Frequency Sweep Test

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Overview

The frequency sweep is the most significant test for polymer melt characterization. In a frequency sweep, the frequency is varied linearly or logarithmically. Strain, stress, and phase are recorded. The strain amplitude of the sinusoidal deformation remains constant. The dynamic moduli G' (storage) and G'' (loss) are calculated from the sinusoidal stress response. The complex viscosity, often used instead of the complex modulus is the complex modulus divided by the applied frequency. Frequency tests are usually performed in the linear region (small strain)—in this case the results obtained are independent of the applied strain.

For viscoelastic fluids including polymer melts, the dynamic moduli are dependent on the frequency. With decreasing frequency the material behaves more and more viscous. The storage modulus decreases from the frequency independent rubbery plateau to the terminal region with the frequency squared. The loss modulus, dominated by G'' in the rubbery region, decreases much slower, crosses G' at $\tan \delta=1$, and determines the material flow behavior in the terminal region. Since G'' decreases linear with the angular frequency in the terminal region, the complex viscosity, the ratio of modulus and frequency, becomes independent of frequency and reaches a plateau value: the zero shear viscosity.

Materials with a yield stress, gels or cross-linked rubbers do not exhibit a terminal region. The modulus remains mostly independent of frequency. The storage modulus is larger than the loss modulus; the material behaves elastic. Only if the strain amplitude is too high, the material structure, responsible for the energy storage mechanisms disappears; the viscous deformation prevails and the materials exhibits now a pronounced terminal region. For non-linear testing at high strain amplitude, the magnitude and the phase up to the 9th harmonic can be evaluated.

The Oscillation Frequency Sweep test makes a series of dynamic mechanical measurements over a range of frequencies, or at a set of selected frequencies, while holding a constant oscillation amplitude and a constant temperature command. The amplitude can be chosen by a specified strain or strain rate.

Suggested Use

Frequency sweeps are typically used to measure the frequency dependence (and equivalently time-dependence) of the viscoelastic properties of a material. In general, high frequencies correspond to short time scales, and low frequencies correspond to long time scales.

Test Setup

To select an Oscillation Frequency test, see [Using Experimental Procedures](#) for detailed instructions.

When you perform an Oscillation Frequency Sweep test, the following parameters need to be chosen.

Environmental Control

Select the following environmental control parameters:

1. **Temperature:** The temperature selection to maintain throughout the test. Enter the desired test temperature, or select **Inherit Value** to maintain the previously specified temperature at the start of this step. The temperature range is dependent on the configuration of your instrument and the installed environment control system.
2. **Soak time:** The amount of time to delay data acquisition at the start of the step, typically to allow for temperature equilibration. This time is measured from the start of the step if **Wait for temperature** is not selected, or from the point at which the measured temperature becomes stabilized at the specified **Temperature**, if **Wait for temperature** is selected. Because of the mass of the sample, test fixtures, and environmental control systems a "soak time" (*i.e.*, time to equilibrate at temperature) is recommended, particularly when starting steps at different temperatures. A five-minute soak time is sufficient for most samples in cases where the change in temperature is not too large. This time is also used at each increment temperature.
3. **Wait for Temperature:** Select this option to wait until the measured sample temperature reaches the specified temperature before beginning data acquisition. If you wish to begin data collection without waiting for the sample temperature to reach the specified value, disable this option.

Test Parameters

Set up the following test parameters:

Select between **Strain**, **Strain%** or **Strain rate**. The specified strain value can be modified during a test by the presence of an AutoStrain option in a preceding conditioning procedure.

- **Strain:** The strain is defined as the specified amplitude of the strain to be applied to the sample at each measurement. This value is used, along with the sample **geometry** and dimensions, to calculate the peak angular deflection or linear displacement to apply to the sample during the measurement. The strain can be selected to simulate real-life end-use conditions, or it can be a value selected to be within the linear viscoelastic range of the sample, and still provide a large enough signal to ensure good data. Strain may be entered also as a percentage.
- **Strain rate:** This parameter is defined as the specified amplitude of the of the shear rate to be applied to the sample at each measurement. This value is used, along with the test frequency, sample **geometry** and dimensions, to calculate the peak angular deflection or linear displacement to apply to the sample during measurement. Running the test with a *fixed* strain rate has the advantage of maintaining a more uniform torque or force across the range of frequencies used for fluids that are close to Newtonian in behavior.

Frequency Sweep Parameters

Three types of frequency sweeps can be run. Choose the desired method from the list below. The available frequency range is dependent on your instrument type and configuration.

- **Logarithmic sweep:** The logarithmic sweep uses the entered values as the starting and ending range of the test, with intermediate points spaced logarithmically. Logarithmic sweeps can be run in ascending or descending order.
 - Choose between **Frequency (Hz)** or **Angular frequency (rad/s)**, then enter the desired range.
 - Enter the desired **Points per decade**. This sets the number of points collected in each decade, based on the initial value. The final value is always collected, regardless of whether it is part of the normal pattern. [Click here for an example.](#)
- **Linear sweep :**The linear sweep uses the entered values as the starting and ending range of the test, with intermediate points calculated by adding or subtracting the increment until the final value is reached.

- Choose between **Frequency (Hz)** or **Angular frequency (rad/s)**, then enter the desired frequency values.
- Choose between **Increment** or **Number of points** and enter the desired increment method:
 - ❖ **Increment:** The frequency increment specifies the change in frequency between subsequent measurements. If the final frequency is greater than the initial frequency, then this value is added at each measurement. If the final frequency is less than the initial value, then this value is subtracted. The final frequency is always measured, regardless of whether it is generated by the frequency increment or not. [Click here for an example.](#)
 - ❖ **Number of points:** The number of points determines the total number of frequencies collected between the specified frequency range. This includes both the initial and final frequency. [Click here for an example.](#)
- **Discrete frequency sweep:** The discrete frequency sweep takes a measurement at each frequency in a list of frequency values, with up to 10 discrete frequencies being specified. The test frequencies in the list can be run in any order (*i.e.*, do not have to be monotonic).

If you want to edit the values already displayed in the table, place the cursor in the table at the desired point and make your editing changes. The entries will be used in order until the first 0.00 entry is encountered, which is recognized as the end of the table.

- Select **Add** to add a new value at the selected location, shifting all of the other table entries down one position.
- Select **Delete** to remove the selected value, shifting all of the table values below the deleted entry up one position.
- Select **Reset** to clear all of the table values, leaving only one entry.

Data Acquisition

There are additional data collection options that can be adjusted to control how data is obtained and what additional information is collected during the measurement. To access these options, click on the “arrow” to display these test fields.

1. Select the desired **Acquisition Mode**:

- **Correlation Parameters:** These options control how data is sampled for use in the dynamic mechanical correlation used to generate the oscillatory data. These settings are typically used to modify the amount of time for the sample to reach steady state oscillation, and to control the amount of raw data sampled for use in the correlation.
- **Transient:** These options control how the raw data are sampled and saved for external evaluation. The settings are typically used to modify the amount of time for the sample to reach steady state oscillation, and to control the amount of raw data sampled

2. Select the applicable parameters:

- **Points per cycle** [Transient Mode only]: The number of raw data sampled during one period of oscillation.
- **Delay cycles:** The number of cycles (whole or fractional) to apply the sinusoidal deformation at the specified amplitude and frequency, before beginning data collection.
- **Delay time:** The amount of time, in seconds, to apply the sinusoidal deformation at the specified amplitude and frequency before beginning data collection.

Both **Delay cycles** and **Delay time** are used to specify the amount of time used to reach steady state at each measurement. If both Cycles and Time are specified, the longer of the two time periods is used. This can be particularly useful in the case of a frequency sweep, where, at low

frequencies (with very long cycle periods) the delay is determined by the delay cycles, but at higher frequencies the delay can be based on the delay time.

3. **Sampling cycles:** The number of cycles to use for collection of raw data, in units of half cycles. The larger the number, the more raw data is collected for the measurement, but the longer the time required for each data point.
4. **Frequency based correlation:** Select this option to use the instrument correlation default settings for the number of sampling cycles. The number of cycles to use for collection of raw data changes as a function of the selected frequency. With increasing frequency, the number of correlation cycles increases. (<1Hz, 2hcycl; <2, 14hcycl;<4Hz, 30hcycl;<8Hz, 60hcycl;<16Hz, 120hcycl;<32Hz, 240hcycl;<64Hz, 500hcycl;<100Hz, 1000hcycl).
5. **Save waveform** [Correlation Mode only]: Select this option to store a snapshot of the data correlation buffer used for the calculation of the oscillatory data along within the data file for future recall. Note that this waveform snapshot is a small subset of the actual data used in the calculation, and can be used to provide insight into the quality of the stress and strain signals used based on the shape of the waveforms and noise levels present.

See Also

[Viewing the Point Graph](#)

6. **Save image:** Select to store images of the test within the data file for future recall when using the Camera accessory.
7. **Iterative Strain Adjustment:** Use this feature to compensate for system compliance, which can reduce the motor strain applied (command strain) considerably. The effective (sample) strain typically becomes smaller than the applied motor strain when the sample stiffness increases as a result of decreasing temperature or increasing frequency. If this feature is selected, the instrument compares the effective strain with the command strain after 1 cycle of oscillation. If the effective strain is outside of the range defined by the tolerance, the motor strain is adjusted assuming constant modulus. The adjustment continues until the effective strain is within the tolerance, the maximum number of attempts to adjust the strain is obtained, or the transducer gives an overload error. Note that the motor does not stop between the strain adjustment cycles and as such, the Axial Force control remains inactive until a data point is recorded.
 - **Strain tolerance:** The tolerance in % of the command strain signal.
 - **Maximum attempts:** Maximum repeated adjustment. If maximum number of adjustments is reached, the experiment is continued using the strain obtained in the last adjustment cycle.
8. **Use additional harmonics** [Correlation Mode only]: Select this option to specify that additional correlations be made at the specified harmonics. These calculations result in additional variables such as the harmonic magnitude and phase of the stress and strain signals being added to the test. The presence of harmonics is due to nonlinearities in the sample response.

Step Termination

TRIOS Software allows you to define conditions in which a step is halted ahead of its normal termination conditions (Limit checking). 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.

Test mode steps can be conditionally terminated by checking the limit or the equilibrium of test variables. The procedure will continue with the next procedure step. When a test mode is conditionally terminated, an event log is created with the termination details.

1. Check the box to enable **Limit checking** termination. Select the test variable from the list, the condition (< smaller than or > larger than) and the value.
2. Check the box to enable the **Equilibrium** termination. Select the variable to track for equilibrium, the tolerance in % and how many data points in sequence have to fulfill the criterion before the test step is terminated.

- NOTES:

- When both **Limit checking** and **Equilibrium** conditions are checked, the test is terminated by the condition that comes first.
 - In a Multiwave test the test termination uses the fundamental value of the test variables only (not the harmonic values).
 - The test termination process is not synchronized with test mode. Since a test mode only stops after completion of the current data point acquisition cycle, additional data points can still be collected after the test termination condition has been fulfilled.
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7 Test: Oscillation amplitude sweep

Setting Up an Oscillation Amplitude Sweep

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[Step Termination](#)

Overview

In the **oscillation** amplitude sweep, the strain or stress amplitude is varied linearly or logarithmically. Strain, stress amplitude, and phase shift are recorded. The main reason for the amplitude sweep is to determine the onset of nonlinear behavior. In the nonlinear region the data points are not equally spaced as a function of strain amplitude, when the stress amplitude is controlled. In the nonlinear region, the material response is not sinusoidal anymore, and odd higher harmonics become important. The magnitude and the phase up to the 9th harmonic can be evaluated.

The onset point of nonlinear behavior in a strain sweep is often used to determine the yield behavior. The critical strain is a measure of the yield point, whereas the corresponding stress is the yield stress. Strain sweeps on structured fluids provide meaningful data only, when the sample is in equilibrium prior to testing.

Test Setup

To select an **Oscillation** Amplitude test, see [Using Experimental Procedures](#) for detailed instructions.

When you perform an **Oscillation** Amplitude test, the following parameters need to be chosen.

Environmental Control

Select the following environmental control parameters:

1. **Temperature:** The temperature range is dependent on the configuration of your instrument and the installed environment control system. The start and end temperature, as well as the temperature increment, are dependent on the transition(s) being evaluated. The initial and final temperatures should bracket the entire range of interest. Enter the desired start test temperature or select **Inherit Value** to maintain the current temperature conditions at the start of this step.
2. **Wait for temperature:** Select this option to wait until the entered temperature is reached before beginning the test. If you wish to begin data collection, while achieving the temperature, disable this option.
3. **Soak time:** The amount of time to delay data acquisition at the start of the step, typically to allow for temperature equilibration. This time is measured from the start of the step if **Wait for temperature** is not selected, or from the point at which the measured temperature becomes stabilized at the commanded **Temperature** if **Wait for temperature** is selected. Because of the mass of the sample, geometries and environmental control systems, a "soak time" (i.e., time to equilibrate at temperature) is recommended, particularly when starting experiments at subambient temperatures or when the temperature is changed significantly between steps. A five-minute soak time is sufficient for most samples in cases where the change in temperature is not too large. This time is also used at each increment temperature.

Test Parameters

Set up the following test parameters.

1. Choose between **Frequency (Hz)** and **Angular Frequency (rad/s)**.
2. Three types of **oscillation** amplitude sweep tests can be run. Choose the desired method from the list below. The available range is dependent on your instrument type and configuration.
 - **Logarithmic sweep**: The logarithmic sweep uses the entered values as the starting and ending range of the test, with intermediate points spaced logarithmically. Logarithmic sweeps can be run in ascending or descending order.
 - ❖ Choose between **Strain** or **Strain %** then enter the desired range.
 - ❖ Enter the desired **Points per decade**. This sets the number of points collected in each decade, based on the initial value. The final value is always collected, regardless of whether it is part of the normal pattern. As an example, consider a sweep conducted over a frequency range of 5 and 28 rad/s, with an increment of 5: Frequencies: 5, 10, 15, 20, 25, 28 rad/s.
 - **Linear sweep**: The linear sweep uses the entered values as the starting and ending range of the test, with intermediate points calculated by adding or subtracting the increment until the final value is reached.
 - ❖ Choose between **Strain** and **Strain %** then enter the desired range.
 - ❖ Choose between **Increment** or **Number of points** to be used when collecting test data.
 - ❖ Enter the desired **Increment**: The increment specifies the change in value between subsequent measurements. If the final value is greater than the initial value, then this value is added at each measurement. If the final value is less than the initial value, then this value is subtracted. The final value is always measured, regardless of whether it is generated by the increment or not. As an example, consider a sweep conducted over a frequency range of 5 and 28 rad/s, with an increment of 5: Frequencies: 5, 10, 15, 20, 25, 28 rad/s.
 - ❖ Enter the desired **Number of points**: The number of data collected in the specified range. This includes both the initial and final value. This includes both the initial and final value. As an example, consider a sweep conducted over a single decade of frequency (between 10 and 100 radians per second). Selecting five data points to be measured divides the frequency range into four equally-spaced frequencies, so that five discrete frequencies are generated: Frequencies: 10, 32.5, 55, 77.5, 100 rad/s.
 - **Discrete sweep**: The discrete sweep takes a measurement at each frequency in a list of frequency values, with up to 10 discrete frequencies being specified. The test frequencies in the list can be run in any order (*i.e.*, do not have to be monotonic).
 1. Select between **Frequency (Hz)** or **Angular Frequency (rad/s)**.
 2. Enter the initial frequency into the first field.
 3. Click **Add** to specify another frequency.
 4. Repeat step 2-3 until the desired frequency values are specified.

If you want to edit the values already displayed in the table, place the cursor in the table at the desired point and make your editing changes. The entries will be used in order until the first 0.00 entry is encountered, which is recognized as the end of the table.

- Use the **Add** button to add a new value at the selected location, shifting all of the other table entries down one position.
- Use the **Remove** button to remove the selected value, shifting all of the table values below the deleted entry up one position.
- Use the **Reset** button to clear all of the table values, leaving only one entry.

Data Acquisition

There are additional data collection options that can be adjusted to control how data is obtained and what additional information is collected during the measurement. To access these options, click on the arrow to display these test fields.

1. Select the desired **Acquisition Mode**:

- **Correlation**: These options control how data is sampled for use in the dynamic mechanical correlation used to generate the oscillatory data. These settings are typically used to modify the amount of time for the sample to reach steady state **oscillation**, and to control the amount of raw data sampled for use in the correlation.
- **Transient**: These options control how the raw data are sampled and saved for external evaluation. The settings are typically used to modify the amount of time for the sample to reach steady state **oscillation**, and to control the amount of raw data sampled.

2. Select the applicable parameters:

- **Points per cycle** [Transient Mode only]: The number of raw data sampled during one period of **oscillation**.
- **Delay cycles**: The number of cycles (whole or fractional) to apply the sinusoidal deformation at the specified amplitude and frequency, before beginning data collection.
- **Delay time**: The amount of time, in seconds, to apply the sinusoidal deformation at the specified amplitude and frequency before beginning data collection.

Both **Delay cycles** and **Delay time** are used to specify the amount of time used to reach steady state at each measurement. If both Cycles and Time are specified, the longer of the two time periods is used. This can be particularly useful in the case of a frequency sweep, where, at low frequencies (with very long cycle periods) the delay is determined by the delay cycles, but at higher frequencies the delay can be based on the delay time.

3. **Sampling cycles**: Enter the number of half cycles you wish to sample the raw data
4. **Frequency based correlation**: Select this option to use the instrument default settings for the number of sampling cycles. The number of cycles to use for collection of raw data changes as a function of the selected frequency. With increasing frequency, the number of correlation cycles increases. (<1Hz, 2hcycl; <2, 14hcycl; <4Hz, 30hcycl; <8Hz, 60hcycl; <16Hz, 120hcycl; <32Hz, 240hcycl; <64Hz, 500hcycl; <100Hz, 1000hcycl)
5. **Save waveform (point display)** [Correlation Mode only]: Select this option to store a snapshot of the data correlation buffer used for the calculation of the oscillatory data along within the data file for future recall. Note that this waveform snapshot is a small subset of the actual data used in the calculation, and can be used to provide insight into the quality of the stress and strain signals used based on the shape of the waveforms and noise levels present.

See Also

[Viewing the Point Display](#)

6. **Save image**: Select to store images of the test within the data file for future recall when using the Camera accessory.
7. **Iterative Strain Adjustment**: Use this feature to compensate for system compliance, which can reduce the motor strain applied (command strain) considerably. The effective (sample) strain typically becomes smaller than the applied motor strain when the sample stiffness increases as a result of decreasing temperature or increasing frequency. If this feature is selected, the instrument compares the effective strain with the command strain after 1 cycle of **oscillation**. If the effective strain is outside of the range defined by the tolerance, the motor strain is adjusted assuming constant modulus. The adjustment continues until the effective strain is within the tolerance, the maximum number of attempts to adjust the strain is obtained, or the transducer gives an overload error. Note that the motor does not stop between the strain adjustment cycles and as such, the Axial Force control remains inactive until a data point is recorded.
- **Strain tolerance**: The tolerance in % of the command strain signal.
 - **Maximum attempts**: Maximum repeated adjustment. If maximum number of adjustments is reached, the experiment is continued using the strain obtained in the last adjustment cycle.

8. **Use additional harmonics** [Correlation Mode only]: Select this option to specify that additional correlations be made at the specified harmonics. These calculations result in additional variables such as the harmonic magnitude and phase of the stress and strain signals being added to the test. The presence of harmonics is due to nonlinearities in the sample response.

Step Termination

TRIOS Software allows you to define conditions in which a step is halted ahead of its normal termination conditions (Limit checking). 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.

Test mode steps can be conditionally terminated by checking the limit or the equilibrium of test variables. The procedure will continue with the next procedure step. When a test mode is conditionally terminated, an event log is created with the termination details.

1. Check the box to enable **Limit checking** termination. Select the test variable from the list, the condition (< smaller than or > larger than) and the value.
2. Check the box to enable the **Equilibrium** termination. Select the variable to track for equilibrium, the tolerance in % and how many data points in sequence have to fulfill the criterion before the test step is terminated.

- NOTES:

- When both **Limit checking** and **Equilibrium** conditions are checked, the test is terminated by the condition that comes first.
- In a Multiwave test the test termination uses the fundamental value of the test variables only (not the harmonic values).
- The test termination process is not synchronized with test mode. Since a test mode only stops after completion of the current data point acquisition cycle, additional data points can still be collected after the test termination condition has been fulfilled.



8 Test: Oscillation cycle sweep

Setting Up an Oscillation Cycle Sweep

In this topic

[Overview](#)
[Suggested Use](#)
[Test Setup](#)
[Environmental Control](#)
[Test Parameters](#)
[Data Acquisition](#)
[Advanced](#)
[Step Termination](#)

Overview

In an **oscillation** cycle sweep, the sample is continuously loaded/unloaded with a cyclic deformation for a selected number of cycles.

Suggested Use

Use this procedure for fatigue testing of solid samples (typically in torsion) under constant strain amplitude and frequency.

Test Setup

To select an **Oscillation** Cycle Sweep test, see [Using Experimental Procedures](#) for detailed instructions.

When you perform an **Oscillation** Cycle Sweep experiment, the following parameters need to be chosen:

Environmental Control

1. **Temperature:** The temperature range is dependent on the configuration of your instrument and the installed environment control system. Enter the desired start test temperature or select **Inherit Value** to maintain the current temperature conditions at the start of this step.
2. **Wait for Temperature:** Select this option to wait until the entered temperature is reached before beginning the test. If you wish to begin data collection while achieving the temperature, disable this option.
3. **Soak Time:** The amount of time to delay data acquisition at the start of the step, typically to allow for temperature equilibration. This time is measured from the start of the step if **Wait for Temperature** is not selected, or from the point at which the measured temperature becomes stabilized at the commanded **Temperature** if **Wait for Temperature** is selected. Because of the mass of the sample, geometries and environmental control systems, a "soak time" (i.e., time to equilibrate at temperature) is recommended, particularly when starting experiments at sub-ambient temperatures or when the temperature is changed significantly between steps. A five-minute soak time is sufficient for most samples in cases where the change in temperature is not too large. This time is also used at each increment temperature.

Test Parameters

Set up the following test parameters:

1. Select the total cycles number. The total cycles and the frequency define the extent of the test. The computed total test time is displayed below.
2. Measure every user selectable number of cycles to determine the complex modulus and the phase.
3. Select between **Strain** and **Strain %**. The specified strain value can be modified during a test by the presence of an AutoStrain option in the Gap Control Panel or a preceding Conditioning procedure.

The strain is defined as the specified amplitude of the strain to be applied to the sample at each measurement. This value is used, along with the sample geometry and dimensions, to calculate the peak angular deflection or linear displacement to apply to the sample during the measurement. The strain selected in a **oscillation** cycle sweep should simulate real-life end-use conditions. Strain may also be entered as a percentage.

4. Select between **Frequency (Hz)** or **Angular Frequency (rad/s)** .
5. Enter the desired frequency into the first field.

Data Acquisition

There are additional data collection options that can be adjusted to control how data is obtained and what additional information is collected during the measurement. To access these options, click on the arrow to display these test fields.

1. Select the desired **Acquisition Mode**:
 - **Correlation**: These options control how data is sampled for use in the dynamic mechanical correlation used to generate the oscillatory data. These settings are typically used to modify the amount of time for the sample to reach steady state **oscillation**, and to control the amount of raw data sampled for use in the correlation.
 - **Transient**: These options control how the raw data are sampled and saved for external evaluation. The settings are typically used to modify the amount of time for the sample to reach steady state **oscillation**, and to control the amount of raw data sampled.
2. Select the applicable parameters:
 - **Points per cycle** [Transient Mode only]: The number of raw data sampled during one period of **oscillation**.
 - **Delay cycles**: The number of cycles (whole or fractional) to apply the sinusoidal deformation at the specified amplitude and frequency, before beginning data collection.
 - **Delay time**: The amount of time, in seconds, to apply the sinusoidal deformation at the specified amplitude and frequency before beginning data collection.

Both **Delay cycles** and **Delay time** are used to specify the amount of time used to reach steady state at each measurement. If both Cycles and Time are specified, the longer of the two time periods is used. This can be particularly useful in the case of a frequency sweep, where, at low frequencies (with very long cycle periods) the delay is determined by the delay cycles, but at higher frequencies the delay can be based on the delay time.

3. **Sampling cycles**: Enter the number of half cycles you wish to sample the raw data
4. **Save waveform (point display)** [Correlation Mode only]: Select this option to store a snapshot of the data correlation buffer used for the calculation of the oscillatory data along within the data file for future recall. Note that this waveform snapshot is a small subset of the actual data used in the calculation, and can be used to provide insight into the quality of the stress and strain signals used based on the shape of the waveforms and noise levels present.

See Also

[Viewing the Point Display](#)

5. **Save image:** Select to store images of the test within the data file for future recall when using the Camera accessory.
 - **Save image every:** Specify the collection rate for time-based experiments. Select the minimum time in seconds between images. An image is stored with the next test point when the time between images has elapsed.
6. **Iterative Strain Adjustment:** Use this feature to compensate for system compliance, which can reduce the motor strain applied (command strain) considerably. The effective (sample) strain typically becomes smaller than the applied motor strain when the sample stiffness increases as a result of decreasing temperature or increasing frequency. If this feature is selected, the instrument compares the effective strain with the command strain after 1 cycle of **oscillation**. If the effective strain is outside of the range defined by the tolerance, the motor strain is adjusted assuming constant modulus. The adjustment continues until the effective strain is within the tolerance, the maximum number of attempts to adjust the strain is obtained, or the transducer gives an overload error. Note that the motor does not stop between the strain adjustment cycles and as such, the Axial Force control remains inactive until a data point is recorded.
 - **Strain tolerance:** The tolerance in % of the command strain signal.
 - **Maximum attempts:** Maximum repeated adjustment. If maximum number of adjustments is reached, the experiment is continued using the strain obtained in the last adjustment cycle.

Step Termination

TRIOS Software allows you to define conditions in which a step is halted ahead of its normal termination conditions (Limit checking). 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.

Test mode steps can be conditionally terminated by checking the limit or the equilibrium of test variables. The procedure will continue with the next procedure step. When a test mode is conditionally terminated, an event log is created with the termination details.

1. Check the box to enable **Limit checking** termination. Select the test variable from the list, the condition (< smaller than or > larger than) and the value.
 2. Check the box to enable the **Equilibrium** termination. Select the variable to track for equilibrium, the tolerance in % and how many data points in sequence have to fulfill the criterion before the test step is terminated.
- NOTES:
 - When both **Limit checking** and **Equilibrium** conditions are checked, the test is terminated by the condition that comes first.
 - In a Multiwave test the test termination uses the fundamental value of the test variables only (not the harmonic values).
 - The test termination process is not synchronized with test mode. Since a test mode only stops after completion of the current data point acquisition cycle, additional data points can still be collected after the test termination condition has been fulfilled.
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9 Test: Oscillation fast sampling sweep

Setting Up an Oscillation Fast Sampling Test

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[Test Setup](#)

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Overview

In an **Oscillation** Fast Sampling experiment, a sinusoidal strain is applied to the sample while the measured torque and angular displacement signals are collected in a continuous buffer and correlated at a rate of up to 100 times per second, independent of the selected test frequency. The size of the buffer is defined by the number of cycles set for the data acquisition. Although the correlation rate is independent of the excitation frequency, the correlation rate should be set to allow at least 20% renewal of the buffer content for each consecutive correlation. Since one data point averages at least 1/2 of a cycle, the test frequency has to be selected such that the **oscillation** period is much smaller than the duration of the reaction process. AutoStrain is not active during fast sampling experiments.

Typical applications are fast curing processes, such as fast structure recoveries after previous shearing.

- NOTE: Transducer autoranging is disabled during this test. Set the desired transducer range using the Transducer Control Panel before starting the experiment.

Test Setup

To select an **Oscillation** Fast Sampling test, see [Using Experimental Procedures](#) for detailed instructions.

When you perform an **Oscillation** Fast Sampling test, the following parameters need to be chosen.

Environmental Control

Select the following environmental control parameters after choosing between **Isothermal** or **Ramp** control:

Isothermal control

1. **Temperature**: The temperature range is dependent on the configuration of your instrument and the installed environment control system. The start and end temperature, as well as the temperature increment, are dependent on the transition(s) being evaluated. The initial and final temperatures should bracket the entire range of interest. Enter the desired start test temperature or select **Inherit value** to maintain the current temperature conditions at the start of this step.
2. **Wait for temperature**: Select this option to wait until the entered temperature is reached before beginning the test. If you wish to begin data collection, while achieving the temperature, disable this option.
3. **Soak time**: The amount of time to delay data acquisition at the start of the step, typically to allow for temperature equilibration. This time is measured from the start of the step if **Wait for temperature** is not selected, or from the point at which the measured temperature becomes stabilized at the commanded **Temperature** if **Wait for temperature** is selected. Because of the mass of the sample, geometries and environmental control systems, a "soak time" (i.e., time to equilibrate at temperature) is recommended, particularly when starting experiments at subambient temperatures or when the temperature is changed

significantly between steps. A five-minute soak time is sufficient for most samples in cases where the change in temperature is not too large. This time is also used at each increment temperature.

Ramp control

1. **Temperature:** The temperature range is dependent on the configuration of your instrument and the installed environment control system. The start and end temperature, as well as the temperature increment, are dependent on the transition(s) being evaluated. The initial and final temperatures should bracket the entire range of interest. Enter the desired start test temperature or select **Inherit value** to maintain the current temperature conditions at the start of this step.
2. **Wait for temperature:** Select this option to wait until the entered temperature is reached before beginning the test. If you wish to begin data collection, while achieving the temperature, disable this option.
3. **Soak time:** The amount of time to delay data acquisition at the start of the step, typically to allow for temperature equilibration. This time is measured from the start of the step if **Wait for temperature** is not selected, or from the point at which the measured temperature becomes stabilized at the commanded **Temperature** if **Wait for temperature** is selected. Because of the mass of the sample, geometries and environmental control systems, a "soak time" (i.e., time to equilibrate at temperature) is recommended, particularly when starting experiments at subambient temperatures or when the temperature is changed significantly between steps. A five-minute soak time is sufficient for most samples in cases where the change in temperature is not too large. This time is also used at each increment temperature.
4. **Ramp rate:** Enter the desired rate of thermal change that the sample material will undergo during the test.
5. **End temperature:** Enter the desired end test temperature.
6. **Soak time after ramp:** Enter the desired time period during which temperature is held at the selected **End temperature** after the ramp has been completed.

Test Parameters

Set up the following test parameters.

1. **Duration:** Sets the duration of the test (**Isothermal** control only).
2. Select the desired method used to collect your data. Only linear sampling is available.
 - **Sampling rate:** This method is preferred when you plan on collecting larger amounts of data. Enter the desired number of data points collected per second.
 - **Sampling interval:** This method is preferred when using low rates of sample collection. Enter the desired number of seconds between each data point.
 - **Sample points:** Enter the total number of sample points you wish to collect over the entire test time.
3. Select between **Strain** and **Strain %**. During the fast sampling mode, the specified strain value can not be modified during a test using the AutoStrain option in the Gap Control Panel or a preceding Conditioning procedure. The strain is defined as the specified amplitude of the strain to be applied to the sample at each measurement. This value is used, along with the sample geometry and dimensions, to calculate the peak angular deflection or linear displacement to apply to the sample during the measurement.
4. Enter the desired **Single Point** frequency.
 - a. Select between **Frequency** (Hz) or **Angular frequency** (rad/s).
 - b. Enter the desired frequency into the first field.

Data Acquisition

There are additional data collection options that can be adjusted to control how data is obtained and what additional information is collected during the measurement. To access these options, click the **Data acquisition** drop-down arrow.

1. **Correlation Parameters:** These options control how data is sampled for use in the dynamic mechanical correlation used to generate the oscillatory data. These settings are typically used to modify the amount of time for the sample to reach steady state **oscillation**, and to control the amount of raw data sampled for use in the correlation.
 - **Delay cycles:** The number of cycles (whole or fractional) to apply the sinusoidal deformation at the specified amplitude and frequency, before beginning data collection.
 - **Delay time:** The amount of time, in seconds, to apply the sinusoidal deformation at the specified amplitude and frequency before beginning data collection.

Both **Delay cycles** and **Delay time** are used to specify the amount of time used to reach steady state at each measurement. If both Cycles and Time are specified, the longer of the two time periods is used. This can be particularly useful in the case of a frequency sweep, where, at low frequencies (with very long cycle periods) the delay is determined by the delay cycles, but at higher frequencies the delay can be based on the delay time.

2. **Sampling cycles:** The number of cycles to use for collection of raw data, in units of half cycles. The larger the number, the more raw data is collected for the measurement, but the longer the time required for each data point. Note that the maximum data collection rate is 100 points per second.
 - NOTE: The actual number of data points is dependent on the test conditions (dynamic frequency and correlation settings). For measured frequencies above 79 rad/second, the maximum fast sampling rate is 100 to 150 points/second. For measured frequencies below 79 rad/second, the maximum fast sampling rate is 50 to 100 points/second.
3. **Frequency based correlation:** Select this option to use the instrument correlation default settings. The number of cycles to use for collection of raw data changes as a function of the selected frequency. With increasing frequency, the number of correlation cycles increases. (<1Hz, 2hcycl; <2, 14hcycl;<4Hz, 30hcycl;<8Hz, 60hcycl;<16Hz, 120hcycl;<32Hz, 240hcycl;<64Hz, 500hcycl;<100Hz, 1000hcycl).
 - NOTE: Selecting this option will reduce the maximum sampling rate.

Step Termination

TRIOS Software allows you to define conditions in which a step is halted ahead of its normal termination conditions (Limit checking). 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.

Test mode steps can be conditionally terminated by checking the limit or the equilibrium of test variables. The procedure will continue with the next procedure step. When a test mode is conditionally terminated, an event log is created with the termination details.

1. Check the box to enable **Limit checking** termination. Select the test variable from the list, the condition (< smaller than or > larger than) and the value.

2. Check the box to enable the **Equilibrium** termination. Select the variable to track for equilibrium, the tolerance in % and how many data points in sequence have to fulfill the criterion before the test step is terminated.

- NOTES:

- When both **Limit checking** and **Equilibrium** conditions are checked, the test is terminated by the condition that comes first.
- In a **Multiwave** test the test termination uses the fundamental value of the test variables only (not the harmonic values).
- The test termination process is not synchronized with test mode. Since a test mode only stops after completion of the current data point acquisition cycle, additional data points can still be collected after the test termination condition has been fulfilled.



10 Test: Oscillation temperature sweep

Setting Up an Oscillation Temperature Sweep

In this topic

Overview
Suggested Use
Test Setup
Environmental Control
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Data Acquisition
Step Termination

Overview

In an **oscillation** temperature sweep the temperature is varied in steps. The **oscillation** measurements are performed under isothermal conditions. An temperature equilibrium time can be set to make sure isothermal conditions are fulfilled before doing the measurements. In a temperature ramp, the frequency is typically a constant, while the temperature step test lends itself to do multiple frequencies. As such it is possible vary the frequency linearly, logarithmically, or in discrete steps. Strain, stress amplitude and phase shift are recorded.

Suggested Use

The main application for the temperature sweep is the frequency temperature superposition to extend the experimental frequency range. Viscoelastic materials change from solid-like to fluid-like behavior over a frequency or time range of 10 decades and more. This frequency range is experimentally not accessible, and can only be constructed from frequency sweeps measured over a wide temperature range. The composite curve referenced to one temperature is the mastercurve.

Similar to the time sweep, the AxialForce functions to compensate for volume changes due to temperature and the AutoStrain function to keep the torque in the instrument operation range are available.

Test Setup

To select an **Oscillation** Temperature Sweep test, see [Using Experimental Procedures](#) for detailed instructions.

When you perform an **Oscillation** Temperature Sweep experiment, the following parameters need to be chosen:

Environmental Control

1. **Start temperature:** The temperature range is dependent on the configuration of your instrument and the installed environment control system. The start and end temperature, as well as the temperature increment, are dependent on the transition(s) being evaluated. The initial and final temperatures should bracket the entire range of interest. Enter the desired start test temperature or select **Inherit Value** to maintain the current temperature conditions at the start of this step.
2. **Wait for temperature:** Select this option to wait until the entered temperature is reached before beginning the test. If you wish to begin data collection, while achieving the temperature, disable this option.
3. **Soak time:** The amount of time to delay data acquisition at the start of the step, typically to allow for temperature equilibration. This time is measured from the start of the step if **Wait for temperature** is not selected, or from the point at which the measured temperature becomes stabilized at the commanded

Temperature if **Wait for temperature** is selected. Because of the mass of the sample, geometries and environmental control systems, a "soak time" (i.e., time to equilibrate at temperature) is recommended, particularly when starting experiments at subambient temperatures or when the temperature is changed significantly between steps. A five-minute soak time is sufficient for most samples in cases where the change in temperature is not too large. This time is also used at each increment temperature.

4. **End temperature:** Enter the desired end test temperature.
5. **Temperature step:** The temperature increment should be small enough to provide good resolution of events. Typically, 3 to 10°C increment is used for TTS. If a two-decade wide frequency is selected, then the increment can be as high as 10°C.

Test Parameters

Set up the following test parameters:

- Select between **Strain**, or **Strain %**. The specified strain value can be modified during a test by the presence of an AutoStrain option in the Gap Control Panel or a preceding Conditioning procedure.

The strain is defined as the specified amplitude of the strain to be applied to the sample at each measurement. This value is used, along with the sample geometry and dimensions, to calculate the peak angular deflection or linear displacement to apply to the sample during the measurement. The strain can be selected to simulate real-life end-use conditions, or it can be a value selected to be within the linear viscoelastic range of the sample, and still provide a large enough signal to ensure good data. Strain may be entered also as a percent.

Frequency Sweep Parameters

Three types of frequency sweeps can be run. Choose the desired method from the list below. The available frequency range is dependent on your instrument type and configuration.

- **Single point**
 - a. Choose between **Angular Frequency** or **Frequency**.
 - b. Enter the initial value into the first field.
- **Logarithmic Sweep:** The logarithmic sweep uses the entered values as the starting and ending range of the test, with intermediate points spaced logarithmically. Logarithmic sweeps can be run in ascending or descending order.
 - a. Choose between **Strain**, or **Strain %**
 - b. Enter the desired range.
 - c. Enter the desired **Points per Decade**. This sets the number of points collected in each decade, based on the initial value. The final value is always collected, regardless of whether it is part of the normal pattern.
- **Linear Sweep:** The linear sweep uses the entered values as the starting and ending range of the test, with intermediate points calculated by adding or subtracting the increment until the final value is reached.
 - a. Choose between **Frequency (Hz)** or **Angular Frequency (rad/s)** then enter the desired frequency values.
 - b. Choose the desired increment method:
 - ❖ **Increment:** The frequency increment specifies the change in frequency between subsequent measurements. If the final frequency is greater than the initial frequency, then this value is added at each measurement. If the final frequency is less than the initial value, then this value is subtracted. The final frequency is always measured, regardless of whether it is generated by the frequency increment or not. [Click here for an example.](#)
 - ❖ **Number of points:** The number of points determines the total number of frequencies collected between the specified frequency range. This includes both the initial and final frequency.

- **Discrete Frequency Sweep:** The discrete frequency sweep takes a measurement at each frequency in a list of frequency values, with up to 10 discrete frequencies being specified. The test frequencies in the list can be run in any order (i.e., do not have to be monotonic).
 - a. Choose between **Angular Frequency** or **Frequency**.
 - b. Enter the initial value into the first field.
 - c. Click **Add** to specify another time or use the down arrow to move to the next entry.
 - d. Repeat step 2-3 until the desired values are specified.

If you want to edit the values already displayed in the table, place the cursor in the table at the desired point and make your editing changes. The entries will be used in order until the first 0.00 entry is encountered, which is recognized as the end of the table.

- Use the **Add** button to add a new value at the selected location, shifting all of the other table entries down one position.
- Use the **Remove** button to remove the selected value, shifting all of the table values below the deleted entry up one position.
- Use the **Reset** button to clear all of the table values, leaving only one entry.

Data Acquisition

There are additional data collection options that can be adjusted to control how data is obtained and what additional information is collected during the measurement. To access these options, click on the “arrow” to display these test fields.

1. Select the desired **Acquisition Mode**:

- **Correlation Parameters:** These options control how data is sampled for use in the dynamic mechanical correlation used to generate the oscillatory data. These settings are typically used to modify the amount of time for the sample to reach steady state **oscillation**, and to control the amount of raw data sampled for use in the correlation.
- **Transient:** These options control how the raw data are sampled and saved for external evaluation. The settings are typically used to modify the amount of time for the sample to reach steady state **oscillation**, and to control the amount of raw data sampled.

2. Select the applicable parameters:

- **Points per cycle** [Transient Mode only]: The number of raw data sampled during one period of **oscillation**.
- **Delay cycles:** The number of cycles (whole or fractional) to apply the sinusoidal deformation at the specified amplitude and frequency, before beginning data collection.
- **Delay time:** The amount of time, in seconds, to apply the sinusoidal deformation at the specified amplitude and frequency before beginning data collection.

Both **Delay cycles** and **Delay time** are used to specify the amount of time used to reach steady state at each measurement. If both Cycles and Time are specified, the longer of the two time periods is used. This can be particularly useful in the case of a frequency sweep, where, at low frequencies (with very long cycle periods) the delay is determined by the delay cycles, but at higher frequencies the delay can be based on the delay time.

3. **Sampling cycles:** The number of cycles to use for collection of raw data, in units of half cycles. The larger the number, the more raw data is collected for the measurement, but the longer the time required for each data point.
4. **Frequency based correlation:** Select this option to use the instrument correlation default settings for the number of sampling cycles. The number of cycles to use for collection of raw data changes as a function of the selected frequency. With increasing frequency, the number of correlation cycles

increases. (<1Hz, 2hcycl; <2, 14hcycl;<4Hz, 30hcycl;<8Hz, 60hcycl;<16Hz, 120hcycl;<32Hz, 240hcycl;<64Hz, 500hcycl;<100Hz, 1000hcycl)

5. **Save waveform:** Select this option to store a snapshot of the data correlation buffer used for the calculation of the oscillatory data along within the data file for future recall. Note that this waveform snapshot is a small subset of the actual data used in the calculation, and can be used to provide insight into the quality of the stress and strain signals used based on the shape of the waveforms and noise levels present.
6. **Save image:** Select to store images of the test within the data file for future recall when using the Camera accessory.
7. **Iterative Strain Adjustment:** Use this feature to compensate for system compliance, which can reduce the motor strain applied (command strain) considerably. The effective (sample) strain typically becomes smaller than the applied motor strain when the sample stiffness increases as a result of decreasing temperature or increasing frequency. If this feature is selected, the instrument compares the effective strain with the command strain after 1 cycle of **oscillation**. If the effective strain is outside of the range defined by the tolerance, the motor strain is adjusted assuming constant modulus. The adjustment continues until the effective strain is within the tolerance, the maximum number of attempts to adjust the strain is obtained, or the transducer gives an overload error. Note that the motor does not stop between the strain adjustment cycles and as such, the Axial Force control remains inactive until a data point is recorded.
 - **Strain tolerance:** The tolerance in % of the command strain signal.
 - **Maximum attempts:** Maximum repeated adjustment. If maximum number of adjustments is reached, the experiment is continued using the strain obtained in the last adjustment cycle.
8. **Use additional harmonics** [Correlation Mode only]: Select this option to specify that additional correlations be made at the specified harmonics. These calculations result in additional variables such as the harmonic magnitude and phase of the stress and strain signals being added to the test. The presence of harmonics is due to nonlinearities in the sample response.

Step Termination

TRIOS Software allows you to define conditions in which a step is halted ahead of its normal termination conditions (Limit checking). 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.

Test mode steps can be conditionally terminated by checking the limit or the equilibrium of test variables. The procedure will continue with the next procedure step. When a test mode is conditionally terminated, an event log is created with the termination details.

1. Check the box to enable **Limit checking** termination. Select the test variable from the list, the condition (< smaller than or > larger than) and the value.
2. Check the box to enable the **Equilibrium** termination. Select the variable to track for equilibrium, the tolerance in % and how many data points in sequence have to fulfill the criterion before the test step is terminated.

- NOTES:

- When both **Limit checking** and **Equilibrium** conditions are checked, the test is terminated by the condition that comes first.
- In a Multiwave test the test termination uses the fundamental value of the test variables only (not the harmonic values).
- The test termination process is not synchronized with test mode. Since a test mode only stops after completion of the current data point acquisition cycle, additional data points can still be collected after the test termination condition has been fulfilled.



11 Test: Oscillation Temperature Ramp

Setting Up an Oscillation Temperature Ramp

In this topic

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Overview

The **oscillation** temperature ramp provides information of how a material changes with temperature. In a temperature ramp the temperature is varied continuously. Typically the frequency is a constant, however, multiple frequencies are possible. In this case the different frequencies are executed consecutively. For simultaneous frequency applications, the Multiwave test mode has to be used since the raw data for all are measured at the same time and temperature. Strain, stress amplitude, and phase shift are recorded. The most important application of the temperature ramp test is the measurement of thermal transition in materials or to simulate cure cycles of thermosetting materials such as structural adhesives, powder coatings, etc.

The most characteristic and important thermal transition in amorphous materials is the glass transition. The glass temperature defines the transition from a solid to a melt. The complex modulus can drop by several decades at T_g . At the glass transition, the thermal expansion coefficient changes dramatically. To compensate for the change in sample shape the Axial Force feature needs to be set in a preceding [conditioning step](#).

Suggested Use

Evolving materials are usually characterized under isothermal condition in the time sweep test, however, industrial processes often operate under non-isothermal conditions. In the temperature ramp, the temperature is ramped up and down to simulate cure operations. Important processing parameters to be determined are the viscosity minimum and the gel point. Both parameters are strongly material and process dependent, and are used for material control as well as for process evaluation.

Similar to the time sweep, the strain can be changed using the Axial Force function to keep the stress within the operation range of the instrument. Care has to be taken controlling the maximum strain. Most reactive systems are complex and transition to non-linear behavior at small total strains already.

Test Setup

To select an **Oscillation** Temperature Ramp test, see [Using Experimental Procedures](#) for detailed instructions.

When you perform an **Oscillation** Temperature Ramp test, the following parameters need to be chosen.

Environmental Control

1. **Start temperature:** The temperature range is dependent on the configuration of your instrument and the installed environment control system. The start and end temperature, as well as the temperature ramp rate, are dependent on the transition(s) being evaluated. The initial and final temperatures should bracket the entire range of interest. Enter the desired start test temperature or select **Inherit value** to maintain the current temperature conditions at the start of this step.

2. **Wait for temperature:** Select this option to wait until the entered temperature is reached before beginning data collection. If you wish to begin data collection, while achieving the temperature, disable this option.
3. **Soak time:** The amount of time to delay data acquisition at the start of the step, typically to allow for temperature equilibration. This time is measured from the start of the step if **Wait for temperature** is not selected, or from the point at which the measured temperature becomes stabilized at the commanded **Temperature** if **Wait for temperature** is selected. Because of the mass of the sample, geometries and environmental control systems, a "soak time" (i.e., time to equilibrate at temperature) is recommended, particularly when starting experiments at subambient temperatures or when the temperature is changed significantly between steps. A five-minute soak time is sufficient for most samples in cases where the change in temperature is not too large. This time is also used at each increment temperature.
4. **Ramp rate:** Enter the desired rate of thermal change that the sample material will undergo during the test.
5. **End temperature:** Enter the desired end test temperature.
6. **Soak time after ramp:** Enter the desired time period during which temperature is held at the selected End Temperature after the ramp has been completed.

Test Parameters

Set up the following test parameters:

1. Select the desired method used to collect your data.
 - **Sampling rate:** This method is preferred when you plan on collecting larger amounts of data. Enter the desired number of data points collected per second.
 - **Sampling interval:** This method is preferred when using low rates of sample collection. Enter the desired number of seconds between each data point.
 - **Number of points:** Enter the total number of sample points you wish to collect over the entire test time.
- NOTE: Assuming **Axial Force**, **AutoStrain**, **Correlation delay**, and **Use additional harmonics** are off, the minimum time (in seconds) per data point required is:

0.1 Hz	1 Hz	10 Hz	100 Hz
20 s	2 s	0.4 s	0.1 s

2. Select between **Strain**, or **Strain %**. The specified strain value can be modified during a test by the presence of an AxialForce option in the Gap Control Panel or a preceding Conditioning procedure.

The strain is defined as the specified amplitude of the strain to be applied to the sample at each measurement. This value is used, along with the sample geometry and dimensions, to calculate the peak angular deflection or linear displacement to apply to the sample during the measurement. The strain can be selected to simulate real-life end-use conditions, or it can be a value selected to be within the linear viscoelastic range of the sample, and still provide a large enough signal to ensure good data. Strain may be entered also as a percent.

Frequency Sweep Parameters

Four types of frequency sweeps can be run. Choose the desired method from the list below. The available frequency range is dependent on your instrument type and configuration.

- **Single point**
 - a. Choose between **Angular Frequency** or **Frequency**.
 - b. Enter the initial value into the first field.

- **Logarithmic sweep:** The logarithmic sweep uses the entered values as the starting and ending range of the test, with intermediate points spaced logarithmically. Logarithmic sweeps can be run in ascending or descending order.
 - a. Choose between **Angular Frequency** or **Frequency**.
 - b. Enter the desired range.
 - c. Enter the desired **Points per decade**. This sets the number of points collected in each decade, based on the initial value. The final value is always collected, regardless of whether it is part of the normal pattern.
- **Linear sweep:** The linear sweep uses the entered values as the starting and ending range of the test, with intermediate points calculated by adding or subtracting the increment until the final value is reached.
 - a. Choose between **Frequency (Hz)** or **Angular Frequency (rad/s)** then enter the desired frequency values.
 - b. Choose the desired increment method:
 - ❖ **Increment:** The frequency increment specifies the change in frequency between subsequent measurements. If the final frequency is greater than the initial frequency, then this value is added at each measurement. If the final frequency is less than the initial value, then this value is subtracted. The final frequency is always measured, regardless of whether it is generated by the frequency increment or not.
 - ❖ **Number of points:** The number of points determines the total number of frequencies collected between the specified frequency range. This includes both the initial and final frequency.
- **Discrete frequency sweep:** The discrete frequency sweep takes a measurement at each frequency in a list of frequency values, with up to 10 discrete frequencies being specified. The test frequencies in the list can be run in any order (i.e., do not have to be monotonic).
 - a. Choose between **Angular frequency** or **Frequency**.
 - b. Enter the initial value into the first field.
 - c. Click **Add** to specify another time or use the down arrow to move to the next entry.
 - d. Repeat step 2-3 until the desired values are specified.

If you want to edit the values already displayed in the table, place the cursor in the table at the desired point and make your editing changes. The entries will be used in order until the first 0.00 entry is encountered, which is recognized as the end of the table.

- Use the **Add** button to add a new value at the selected location, shifting all of the other table entries down one position.
- Use the **Remove** button to remove the selected value, shifting all of the table values below the deleted entry up one position.
- Use the **Reset** button to clear all of the table values, leaving only one entry.

Data Acquisition

There are additional data collection options that can be adjusted to control how data is obtained and what additional information is collected during the measurement. To access these options, click on the "arrow" to display these test fields.

1. Select the desired **Acquisition Mode**:
 - **Correlation Parameters:** These options control how data is sampled for use in the dynamic mechanical correlation used to generate the oscillatory data. These settings are typically used to modify the amount of time for the sample to reach steady state **oscillation**, and to control the amount of raw data sampled for use in the correlation.

- **Transient:** These options control how the raw data are sampled and saved for external evaluation. The settings are typically used to modify the amount of time for the sample to reach steady state **oscillation**, and to control the amount of raw data sampled.

2. Select the applicable parameters:

- **Points per cycle** [Transient Mode only]: The number of raw data sampled during one period of **oscillation**.
- **Delay cycles:** The number of cycles (whole or fractional) to apply the sinusoidal deformation at the specified amplitude and frequency, before beginning data collection.
- **Delay time:** The amount of time, in seconds, to apply the sinusoidal deformation at the specified amplitude and frequency before beginning data collection.

Both **Delay cycles** and **Delay time** are used to specify the amount of time used to reach steady state at each measurement. If both Cycles and Time are specified, the longer of the two time periods is used. This can be particularly useful in the case of a frequency sweep, where, at low frequencies (with very long cycle periods) the delay is determined by the delay cycles, but at higher frequencies the delay can be based on the delay time.

3. **Sampling cycles:** The number of cycles to use for collection of raw data, in units of half cycles. The larger the number, the more raw data is collected for the measurement, but the longer the time required for each data point.
4. **Frequency based correlation:** Select this option to use the instrument correlation default settings for the number of sampling cycles. The number of cycles to use for collection of raw data changes as a function of the selected frequency. With increasing frequency, the number of correlation cycles increases. (<1Hz, 2hcycl; <2, 14hcycl;<4Hz, 30hcycl;<8Hz, 60hcycl;<16Hz, 120hcycl;<32Hz, 240hcycl;<64Hz, 500hcycl;<100Hz, 1000hcycl)
5. **Save waveform:** Select this option to store a snapshot of the data correlation buffer used for the calculation of the oscillatory data along within the data file for future recall. Note that this waveform snapshot is a small subset of the actual data used in the calculation, and can be used to provide insight into the quality of the stress and strain signals used based on the shape of the waveforms and noise levels present.

See Also

[Viewing the Point Display](#)

6. **Save image:** Select to store images of the test within the data file for future recall when using the Camera accessory.
 - **Save image every:** Specify the collection rate for time-based experiments. Select the minimum time in seconds between images. An image is stored with the next test point when the time between images has elapsed.
7. **Iterative Strain Adjustment:** Use this feature to compensate for system compliance, which can reduce the motor strain applied (command strain) considerably. The effective (sample) strain typically becomes smaller than the applied motor strain when the sample stiffness increases as a result of decreasing temperature or increasing frequency. If this feature is selected, the instrument compares the effective strain with the command strain after 1 cycle of **oscillation**. If the effective strain is outside of the range defined by the tolerance, the motor strain is adjusted assuming constant modulus. The adjustment continues until the effective strain is within the tolerance, the maximum number of attempts to adjust the strain is obtained, or the transducer gives an overload error. Note that the motor does not stop between the strain adjustment cycles and as such, the Axial Force control remains inactive until a data point is recorded.
 - **Strain tolerance:** The tolerance in % of the command strain signal.
 - **Maximum attempts:** Maximum repeated adjustment. If maximum number of adjustments is reached, the experiment is continued using the strain obtained in the last adjustment cycle.

8. **Use additional harmonics** [Correlation Mode only]: Select this option to specify that additional correlations be made at the specified harmonics. These calculations result in additional variables such as the harmonic magnitude and phase of the stress and strain signals being added to the test. The presence of harmonics is due to nonlinearities in the sample response.

Step Termination

TRIOS Software allows you to define conditions in which a step is halted ahead of its normal termination conditions (Limit checking). 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.

Test mode steps can be conditionally terminated by checking the limit or the equilibrium of test variables. The procedure will continue with the next procedure step. When a test mode is conditionally terminated, an event log is created with the termination details.

1. Check the box to enable **Limit checking** termination. Select the test variable from the list, the condition (< smaller than or > larger than) and the value.
2. Check the box to enable the **Equilibrium** termination. Select the variable to track for equilibrium, the tolerance in % and how many data points in sequence have to fulfill the criterion before the test step is terminated.

- NOTES:

- When both **Limit checking** and **Equilibrium** conditions are checked, the test is terminated by the condition that comes first.
- In a Multiwave test the test termination uses the fundamental value of the test variables only (not the harmonic values).
- The test termination process is not synchronized with test mode. Since a test mode only stops after completion of the current data point acquisition cycle, additional data points can still be collected after the test termination condition has been fulfilled.



12 Test: Oscillation Time Sweep

Setting Up an Oscillation Time Sweep

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Overview

In an **oscillation** time sweep, the time is the variable. Temperature, frequency, and strain or stress amplitude are typically held constant throughout the test. Strain and stress amplitude, as well as the phase, are recorded to monitor the evolution of a material as a function of time. This test mode can be used to study the stability (thermal, mechanical, etc.) or the evolution of materials with time at constant temperature. As the material evolves, it may be necessary to change the gap due to volume changes using the Axial Force function or to change the strain in order to remain in the instrument torque range using the Axial Force function.

The Axial Force needs to be set in the **Axial Force** panel for pretest control and in a preceding [Conditioning step](#) for control during the test. For fast processes, such as UV curing, use the fast data sampling mode 'Fast sampling - Isothermal' with a sampling up to 500 data pts/s. Since one data point averages at least 1/2 of a cycle, the test frequency has to be selected such that the **oscillation** period is much smaller than the duration of the reaction process.

Suggested Use

For complex materials, the **oscillation** time sweep can be used for pre-testing and to evaluate the effect of strain and time. Since the test parameters frequency and strain can be changed manually during the test via the motor control panels, structure changes can be easily monitored and the testing parameters for **oscillation** frequency and strain sweeps tests validated.

Time sweeps are performed at constant frequency. However, multiple frequencies are possible using the [Multiwave test mode](#). Multiple frequencies are not possible with fast data acquisition.

For non-linear testing at high strain amplitude, the magnitude and the phase up to the 9th harmonic can be evaluated.

Test Setup

To select an **Oscillation** Time Sweep test, see [Using Experimental Procedures](#) for detailed instructions.

When you perform an **Oscillation** Time Sweep experiment, the following parameters need to be chosen:

Environmental Control

1. **Temperature:** The temperature range is dependent on the configuration of your instrument and the installed environment control system. Enter the desired start test temperature or select **Inherit Value** to maintain the current temperature conditions at the start of this step.
2. **Wait for Temperature:** Select this option to wait until the entered temperature is reached before beginning the test. If you wish to begin data collection while achieving the temperature, disable this option.
3. **Soak Time:** The amount of time to delay data acquisition at the start of the step, typically to allow for temperature equilibration. This time is measured from the start of the step if **Wait for Temperature** is not selected, or from the point at which the measured temperature becomes stabilized at the commanded **Temperature** if **Wait for Temperature** is selected. Because of the mass of the sample, geometries and environmental control systems, a "soak time" (i.e., time to equilibrate at temperature) is recommended, particularly when starting experiments at sub-ambient temperatures or when the temperature is changed significantly between steps. A five-minute soak time is sufficient for most samples in cases where the change in temperature is not too large. This time is also used at each increment temperature.

Test Parameters

Set up the following test parameters:

1. Enter the **Duration** time of your experiment.
2. Select the desired method used to collect your data.
 - **Sampling Rate:** This method is preferred when you plan on collecting larger amounts of data. Enter the desired number of data points collected per second.
 - **Sampling interval:** This method is preferred when using low rates of sample collection. Enter the desired number of seconds between each data point.
 - **Number of points:** Enter the total number of sample points you wish to collect over the entire test time.
- Assuming Axial Force, AutoStrain, Correlation delay, and Use additional harmonics are off, the minimum time (in seconds) per data point required is:

0.1 Hz	1 Hz	10 Hz	100 Hz
20 s	2 s	0.4 s	0.1 s

3. Select between **Strain** and **Strain %**. The specified strain value can be modified during a test by the presence of an AutoStrain option in the Gap Control Panel or a preceding Conditioning procedure.

The strain is defined as the specified amplitude of the strain to be applied to the sample at each measurement. This value is used, along with the sample geometry and dimensions, to calculate the peak angular deflection or linear displacement to apply to the sample during the measurement. The strain can be selected to simulate real-life end-use conditions, or it can be a value selected to be within the linear viscoelastic range of the sample, and still provide a large enough signal to ensure good data. Strain may be entered also as a percent.

4. Select between **Frequency (Hz)** or **Angular Frequency (rad/s)**.
5. Enter the desired frequency into the first field.

Data Acquisition

There are additional data collection options that can be adjusted to control how data is obtained and what additional information is collected during the measurement. To access these options, click on the arrow to display these test fields.

1. Select the desired **Acquisition Mode**:
 - **Correlation**: These options control how data is sampled for use in the dynamic mechanical correlation used to generate the oscillatory data. These settings are typically used to modify the amount of time for the sample to reach steady state **oscillation**, and to control the amount of raw data sampled for use in the correlation.
 - **Transient**: These options control how the raw data are sampled and saved for external evaluation. The settings are typically used to modify the amount of time for the sample to reach steady state **oscillation**, and to control the amount of raw data sampled.

2. Select the applicable parameters:

- **Points per cycle** [Transient Mode only]: The number of raw data sampled during one period of **oscillation**.
- **Delay cycles**: The number of cycles (whole or fractional) to apply the sinusoidal deformation at the specified amplitude and frequency, before beginning data collection.
- **Delay time**: The amount of time, in seconds, to apply the sinusoidal deformation at the specified amplitude and frequency before beginning data collection.

Both **Delay cycles** and **Delay time** are used to specify the amount of time used to reach steady state at each measurement. If both Cycles and Time are specified, the longer of the two time periods is used. This can be particularly useful in the case of a frequency sweep, where, at low frequencies (with very long cycle periods) the delay is determined by the delay cycles, but at higher frequencies the delay can be based on the delay time.

3. **Sampling cycles**: Enter the number of half cycles you wish to sample the raw data
4. **Frequency based correlation**: Select this option to use the instrument default settings for the number of sampling cycles. The number of cycles to use for collection of raw data changes as a function of the selected frequency. With increasing frequency, the number of correlation cycles increases. (<1Hz, 2hcycl; <2, 14hcycl;<4Hz, 30hcycl;<8Hz, 60hcycl;<16Hz, 120hcycl;<32Hz, 240hcycl;<64Hz, 500hcycl;<100Hz, 1000hcycl)
5. **Save waveform (point display)** [Correlation Mode only]: Select this option to store a snapshot of the data correlation buffer used for the calculation of the oscillatory data along within the data file for future recall. Note that this waveform snapshot is a small subset of the actual data used in the calculation, and can be used to provide insight into the quality of the stress and strain signals used based on the shape of the waveforms and noise levels present.

See Also

[Viewing the Point Display](#)

6. **Save image**: Select to store images of the test within the data file for future recall when using the Camera accessory.
 - **Save image every**: Specify the collection rate for time-based experiments. Select the minimum time in seconds between images. An image is stored with the next test point when the time between images has elapsed.
7. **Iterative Strain Adjustment**: Use this feature to compensate for system compliance, which can reduce the motor strain applied (command strain) considerably. The effective (sample) strain typically becomes smaller than the applied motor strain when the sample stiffness increases as a result of decreasing temperature or increasing frequency. If this feature is selected, the instrument compares the effective strain with the command strain after 1 cycle of **oscillation**. If the effective strain is outside of the range defined by the tolerance, the motor strain is adjusted assuming constant modulus. The adjustment continues until the effective strain is within the tolerance, the maximum number of attempts to adjust the strain is obtained, or the transducer gives an overload error. Note that the motor does not stop between the strain adjustment cycles and as such, the Axial Force control remains inactive until a data point is recorded.
 - **Strain tolerance**: The tolerance in % of the command strain signal.

- **Maximum attempts:** Maximum repeated adjustment. If maximum number of adjustments is reached, the experiment is continued using the strain obtained in the last adjustment cycle.
8. **Use additional harmonics** [Correlation Mode only]: Select this option to specify that additional correlations be made at the specified harmonics. These calculations result in additional variables such as the harmonic magnitude and phase of the stress and strain signals being added to the test. The presence of harmonics is due to non-linearities in the sample response.

Step Termination

TRIOS Software allows you to define conditions in which a step is halted ahead of its normal termination conditions (Limit checking). 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.

Test mode steps can be conditionally terminated by checking the limit or the equilibrium of test variables. The procedure will continue with the next procedure step. When a test mode is conditionally terminated, an event log is created with the termination details.

1. Check the box to enable **Limit checking** termination. Select the test variable from the list, the condition (< smaller than or > larger than) and the value.
2. Check the box to enable the **Equilibrium** termination. Select the variable to track for equilibrium, the tolerance in % and how many data points in sequence have to fulfill the criterion before the test step is terminated.

- **NOTES:**

- When both **Limit checking** and **Equilibrium** conditions are checked, the test is terminated by the condition that comes first.
- In a Multiwave test the test termination uses the fundamental value of the test variables only (not the harmonic values).
- The test termination process is not synchronized with test mode. Since a test mode only stops after completion of the current data point acquisition cycle, additional data points can still be collected after the test termination condition has been fulfilled.

