

Rheometer Concise Operating Procedures

Please click the links or refer to the section listed for detailed information.

Instruction

1. Instrument Hazards and Best Practices
2. Ensure that instrument has proper air supply. If there is not 30 psi on the regulator behind the instrument, do not touch anything and alert the lab manager.
3. Remove bearing lock, if necessary.
4. Turn on power to instrument, if necessary.
5. Open TRIOS and connect to instrument.
6. Confirm instrument connection to computer.
7. Confirm **instrument** inertia calibration has been performed in the last month. If not, perform calibration. If geometry was already attached and calibration is necessary, remove upper geometry prior to performing calibration.
8. Attach geometry. If geometry was already attached, skip this step.
9. Fix plates to geometry. Ensure you follow the precise procedure in the link.
10. Perform **geometry** inertia calibration.
11. Perform bearing friction correction.
12. Perform rotational mapping.
13. Zero the gap.
14. Load sample.
15. Trim the gap.
16. Go to experimental gap.
17. Set up or load a saved procedure.
18. If performing a temperature sweep, use gap compensation.
19. If performing a cure/thermoset profile, ensure you use the proper safety settings.
20. Run experiment.
21. Clean up and export data files.

Need Help? Contact the lab manager. You can also call the TA helpline for applications support.

Link to detailed info

[Link to Hazards](#)

[2. Air Supply](#)

[3. Bearing Lock](#)

[4. Power Switch](#)

[5. TRIOS](#)

[6. Computer Connection](#)

[7. Instrument Inertia Cal](#)

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Instrument Hazards and Best Practices: Rheometer

This document will cover the inherent hazards when utilizing this piece of equipment as well as the best practices and procedures to avoid danger. These hazards will not include basic things that may be included in the basic safety-training document that each user has attested to have reviewed at fom.wvu.edu/documents

Lab coats are to be provided by the user unless special hazards exist in which case they are located at the PPE station.

Hazards:

- Chemical exposure
- Extreme Temperatures (Up to 600 C)

1. Required PPE

Appropriate laboratory attire is required at all times in the AMSEC laboratories. Whenever chemicals are being used, an additional requirement of a lab coat is required. Lab coats are to be provided by the user.

Whenever a user is in the AMSEC labs, the minimum requirement for eye protection is wrap around impact glasses. Anytime liquid chemicals are present in the same room as the user without a direct barrier, all users in the lab must wear chemical splash goggles. Splash goggles must be approved by State of Washington Administrative Code (WAC 296-155-215).

If chemicals being used are considered toxic, caustic, corrosive, flammable solvents, carcinogenic, mutagenic, or teratogenic, a minimum of disposable nitrile gloves is required. Avoid chemical transfer by taking off gloves when using anything other than the chemical(s).

2. Extreme Temperatures

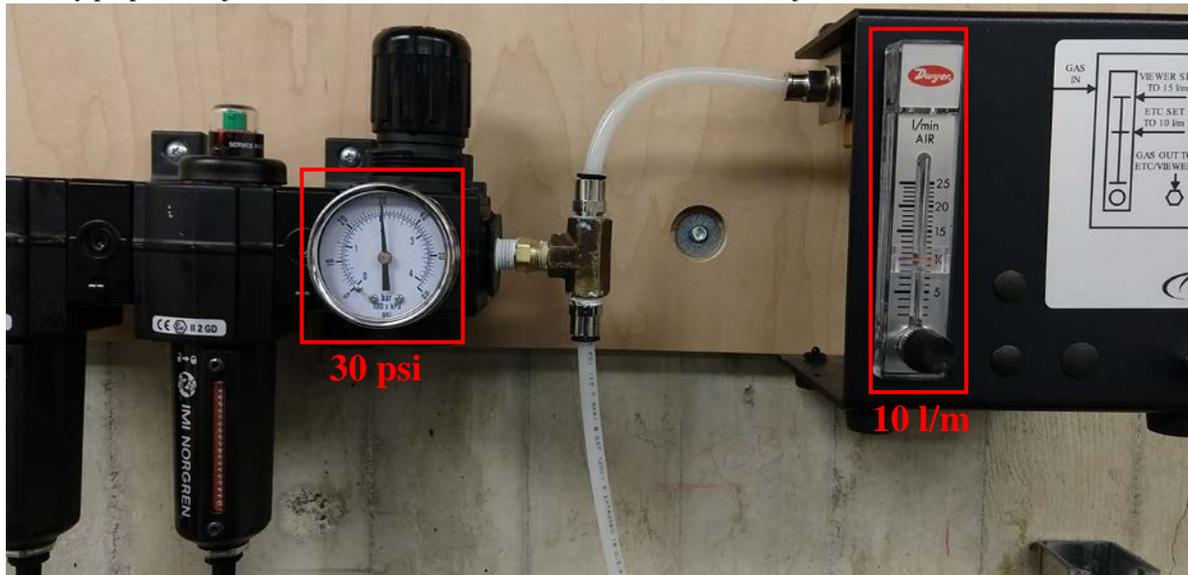
The Rheometer operates from room temperature up to 600 C. Avoid contact with the furnace. As a common practice, users should never touch the interior of the furnace. This is due to not only the temperature risks, but to keep the furnace from becoming contaminated.



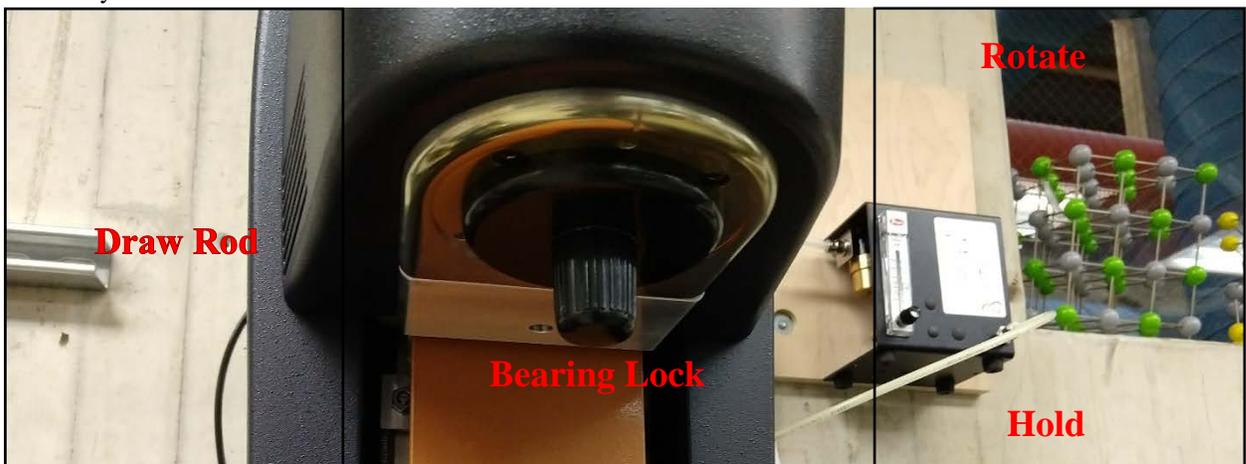
Rheometer Standard Operating Procedures

These sections are not necessarily in order. Please see the [concise operating procedures](#) for logical steps.

1. Ensure computer is on.
2. **Ensure the air supply is on.** Air supply should be at 30 psi for the air bearing. Failure to ensure air supply pressure could result in damage to the radial bearing (\$30k-\$40k). Air supply should never be turned off for any purpose. Adjust flow to environmental test chamber (ETC, this is just the furnace) to 10 l/m if needed.



3. **Remove the black bearing lock,** if necessary, by holding it in place while turning the draw rod knob at the top in a counter-clockwise direction. Once the bearing lock is removed, make sure that the spindle rotates freely.



NOTE: If air supply is interrupted while bearing lock is off, **DO NOT TURN** the **DRIVE SHAFT**; this will cause damage to the radial bearing. Stop immediately and contact the lab manager.

- Turn on the Power to Rheometer, if necessary.
Switch the power button, located on the rear of the electronics control box, to the *ON* position.

Power Switch

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

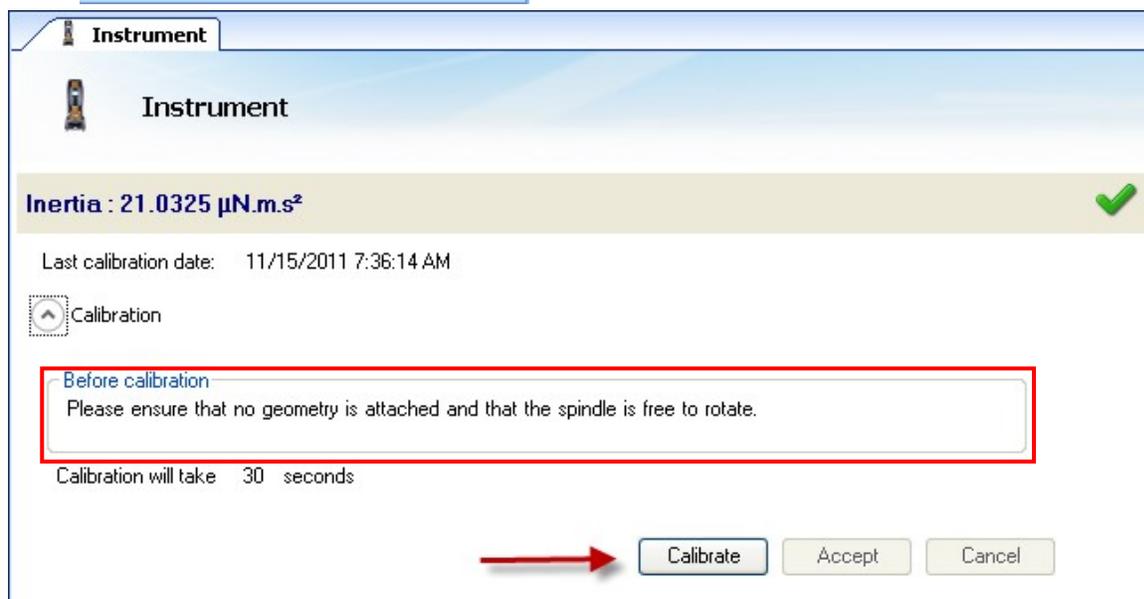
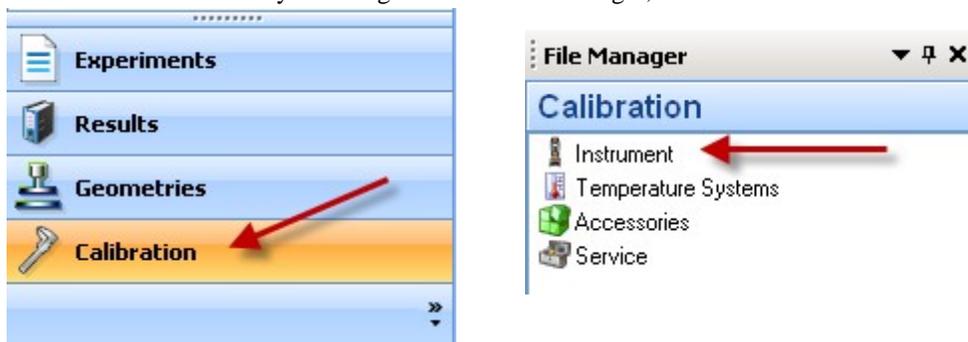
- When the instrument has finished the system check, start the instrument control software, TRIOS, from the desktop. Once the instrument turns green, click “connect”.



- Choose the Control Panel icon from the Home ribbon, , to make certain that communication has been established between the computer and the instrument. If communication is not established, the control panel values will appear undefined. The Control Panel can also be used to manually control the instrument.

7. Instrument Inertia

Determine the instrument inertia by selecting under the File Manager, the *Calibration Tab* and then *Instrument*.

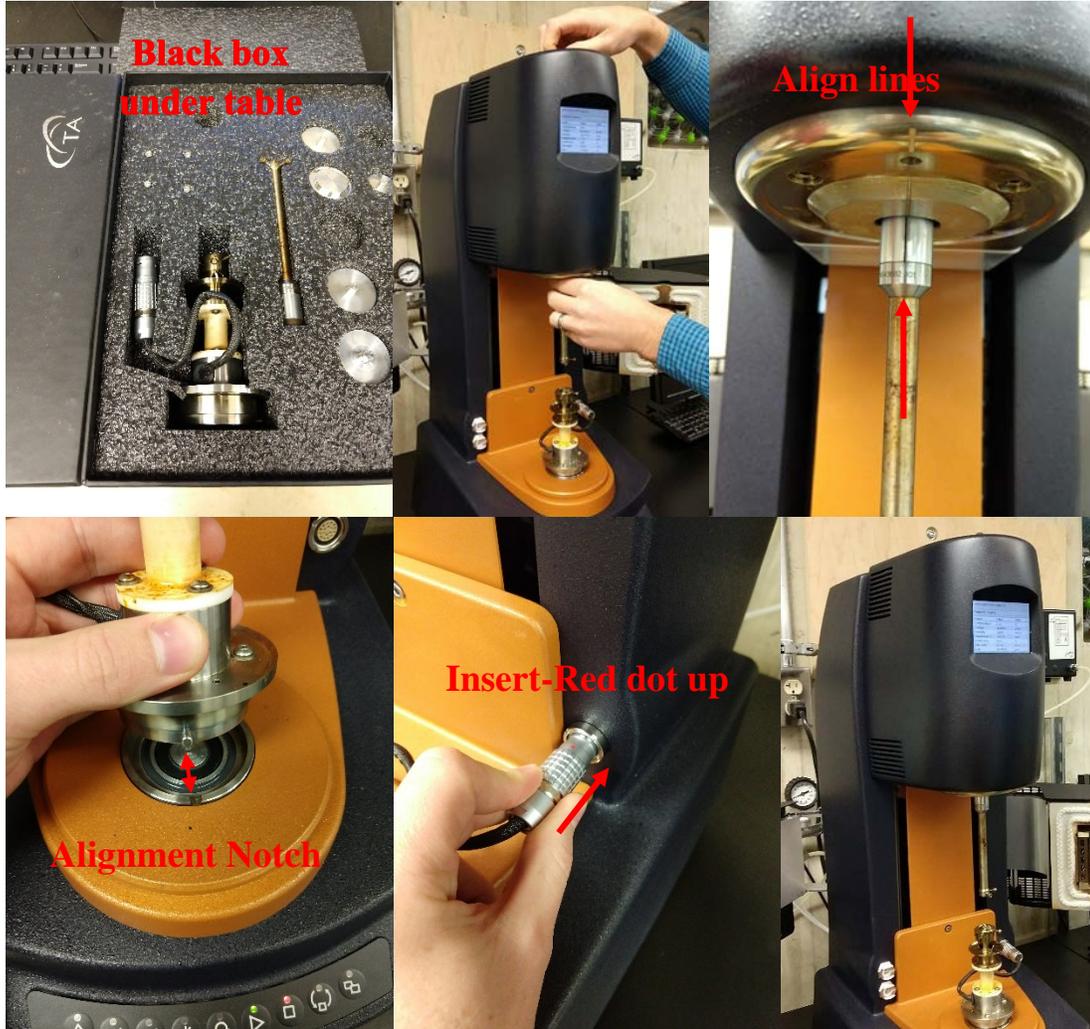


This value is unique for each bearing assembly. An acceptable range for this value is $\sim 21 - 22 \mu\text{Nms}^2$ for the DHR series. The value for the instrument should not change by more than 10% of the original Inertia value.

NOTE: This calibration should be performed on monthly basis.

8. Select a Geometry-25mm Disposable Parallel Plates

Ensure that you have sent the shaft to the home position by holding down the lock button on the Rheometer until you hear a beep. Attach test geometry by sliding it up the drive shaft and hold it stationary while turning the draw rod at the top in a clockwise direction. Line up the line on the geometry with the line on the Rheometer. Attach lower geometry as well.



NOTE: If testing a solid sample, make sure you have spoken to the lab manager beforehand.

9. Attach Disposable Plates

Below the instrument should be the disposable 25mm aluminum plates. The bottom one should have the drip pan and the top one should not. They should be firmly, but not too tightly, attached via the two setscrews on the sides. Be careful not to put lateral pressure on the top shaft to avoid damaging the bearing. After zeroing the gap, double check that the plates are in fact parallel to one another.

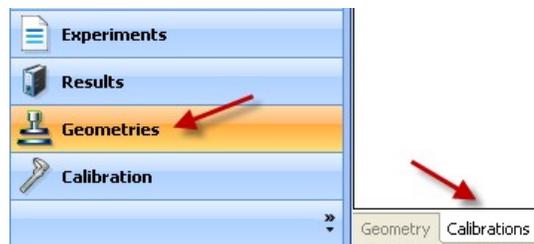
If possible, do not dispose of the plates. Simply clean them with whatever solvent is appropriate. If you do not need to use brand new plates, such as a verification measurement or something that is not as important, please use plates from the box that says clean used plates. New plate sets are \$20 each so we need to try to conserve them as much as possible.

Some people find it is easier to attach the top plate when the geometry is not attached. If you find this to be the case, simply remove the top geometry and attach the top plate before affixing it to the Rheometer.

10. Geometry Inertia

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

Determine the geometry inertia by selecting under the File Manager, the *Geometries Tab*, *Calibrations page* and then *Calibrate*.



 **DIN concentric cylinders, Peltier Steel - 987202**

Inertia : 4.13863 $\mu\text{N.m.s}^2$ ✔

Last calibration date: 11/15/2011 1:55:35 PM

 Calibration

Before calibration

Please ensure that a geometry is attached and that the spindle is free to rotate.

Calibration will take 30 seconds

➔

NOTE: This calibration is required when a new geometry file is first setup. For verification purpose, one can do it any time when changing geometry, but this is not required.

11. Bearing Friction Correction

A magnetic bearing is used to set the drive shaft afloat and provide virtually friction free application of torque to the sample. However, there will always be some residual friction. With most test materials, this is insignificant, but in about 1% of the low viscosity samples, this inherent friction causes inaccuracies in the final rheological data. To overcome this, the software has a bearing friction correction that should be activated. The bearing friction value without any geometry attached is $\sim 0.25 - 0.3 \mu\text{Nm}/(\text{rad/s})$. With attaching a geometry, this value may raise up to about $0.3 - 0.5 \mu\text{Nm}/(\text{rad/s})$. Bearing friction correction can be found just below the Geometry Inertia calibration.



NOTE: Please ensure that the Instrument Inertia (step 7) and Geometry Inertia (step 9) have been calibrated before determining the bearing friction correction value.

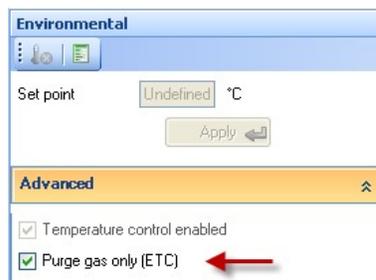
NOTE: When using a pressure cell geometry, this bearing friction value can be as high as $9 - 12 \mu\text{Nm}/(\text{rad/s})$.

NOTE: This calibration is required when a new geometry file is first setup. For verification purpose, one can do it any time when changing geometry, but this is not required.

12. Select a Temperature System

Our Rheometer only has the ETC. If we obtain a low temperature attachment, the following may become useful.

NOTE: If using an ETC equipped with low temperature fittings, checking “purge gas only” (located in the Control Panel → Environmental → Advanced) will convert a low temperature flow meter to use only the ETC purge gas instead of Liquid Nitrogen to control temperature. Recommended lower temperature range when using this setting is 45°C . For tests that require lower temperatures using the ETC, attach LN_2 feed and disable this setting.



13. Zero the Geometry Gap

Choose the zero gap icon located on the Front Control Panel of the DHR, or select GAP from the Control Panel and follow the directions on the screen.



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

NOTE: Zero gap needs to be performed every time.

The gap closure profile options are located in Options → GAP

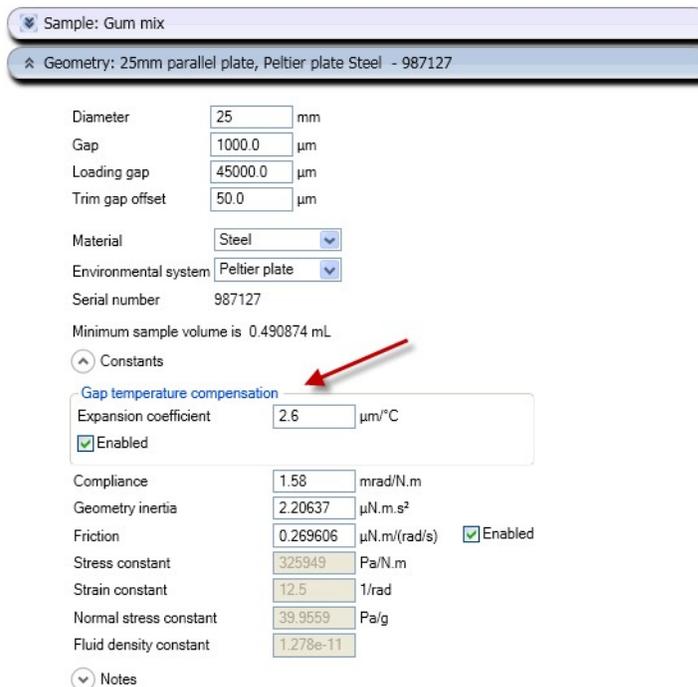
14. Gap Compensation

NOTE: Predetermined values can be entered into the TRIOS software

NOTE: The gap compensation check box must be active, which is located within the Geometry Constants section of the test procedure.

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

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



15. Mapping

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

By combining the absolute angular position data from the optical encoder with microprocessor control of the motor, these small variations can be mapped automatically and stored in memory for subsequent real-time corrections in the test.

To create a mapping, the software rotates the drive shaft at a fixed speed, monitoring the torque required to maintain this speed through a full 360° of rotation. These variations in torque can then be accounted for automatically by the microprocessor, which is in effect carrying out a baseline correction of the torque. This results in a very wide operating range of the bearing without operator intervention – a confidence check in bearing performance.

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

Hold the “Lock” button on the instrument control panel until you hear a beeping. This will lock the drive shaft to its “home” position. Then attach the geometry with the line on the side aligned with the marker on the bottom of the instrument head.



Begin the rotational mapping by going to the Geometries Tab, Calibrations Page, and then choose Rotational Mapping.

Rotational Mapping ⚠

Last calibration date:

⏪ Calibration

Mapping settings

Bearing mapping type ▼
standard

Number of iterations ▼
2

Mapping may take up to 5 minutes to complete.

Options

Display mapping prompt when the geometry changes

Calibrate Cancel

Geometry Calibrations

There are three levels of rotational mapping – fast, standard, and precision. It is also possible to perform multiple mappings iterations. Standard should be good form most applications.

The magnitude of the torque correction varies with the instrument type, but typically has an absolute maximum in the range 0.2 to 1 $\mu\text{N}\cdot\text{m}$. With this in mind, you can determine if mapping will improve your measurements. For example, if the minimum torque during your procedure never drops below 100 $\mu\text{N}\cdot\text{m}$, then the maximum error that an unmapped bearing will contribute is 1%. In this case, you may consider that mapping is not required or that a fast mapping is sufficient. As the torques in your procedure drop, the error from an unmapped bearing increases, so it may become necessary to perform precision mappings to keep these errors to a minimum.

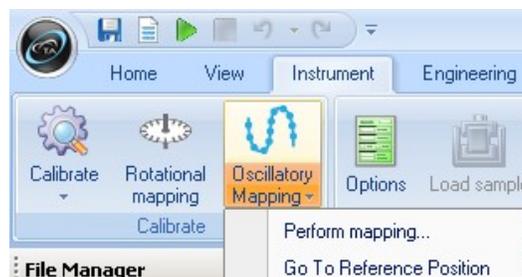
NOTE: This calibration is required when a new geometry file is first setup. The mapping history is recorded in each individual geometry file. If every time the geometry is loaded at the “home” position. There is no need for any additional mapping. But for maximum accuracy at the lower torque end of the rheometer, it is recommended that the mapping be performed each time a new measuring system is used, or if the current geometry is removed for cleaning. We should map every time for disposable plates due to variance between plate sets.

Oscillation Mapping-This mapping will perform a baseline subtraction only when using the continuous controlled strain mode and will improve the performance for low torque, low displacement data. To access in the software, go to the Instrument Ribbon then Oscillation Mapping.

Up to 10 mappings can be stored to disk. These can be either constant amplitude or constant frequency and the software will automatically upload the most appropriate map to the instrument that covers the range of the current test parameters.

NOTE: Check “go to reference position” so that you don’t lose your position of where the mapping was done. Select this option before starting an oscillatory experiment and the experiment would be performed at the position where the mapping was done.

NOTE: This calibration is recommended when performing oscillatory tests on low viscosity samples.



16. Set up a Procedure

16.1. Sample Setup

Fill out the sample section for your experiment. Include as much information as possible. The file name will end up being the “Name” field. Choose a save location appropriate in the Data folder on the C-drive. Please group your data under a research advisor or class folder to avoid cluttering up the main folder.

Sample: Test Sample

Name:

Operator:

Project:

Notes:

Advanced

File Name:

File Path:

Template:

Reset run number when saved

16.2. Geometry within Procedure

There should not need to be any changes in the geometry section unless you want a different loading gap. This is where the volume of sample required to fill the gap can be found.

Geometry: 25mm parallel plate, ETC Aluminium

Diameter: mm

Gap: μm

Loading gap: μm

Trim gap offset: μm

Disposable

Material:

Environmental system:

Minimum sample volume is 0.490874 mL

Constants

Notes

16.3. Experimental Procedure

Create a new procedure by selecting Procedure from the Experiments Tab or open a previously created procedure by selecting the appropriate file. The procedure can be viewed, edited, and adapted in the Procedure segments.

Sample: Gum mix

Geometry: 25mm parallel plate, Peltier plate Steel - 987127

Procedure of 2 steps

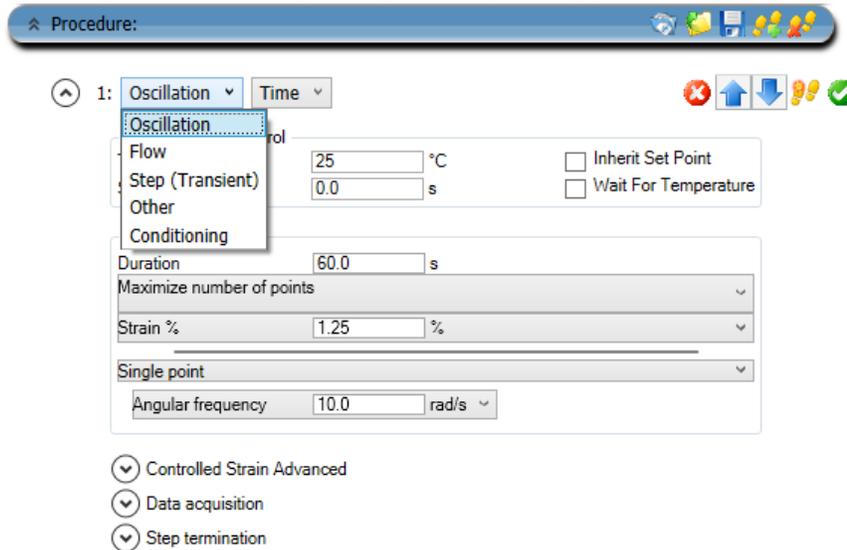
1: Conditioning Sample 25°C

2: Flow Sweep 25°C, 0.01 to 100 1/s

Open saved procedures

For thermosets and cure profiles, please see the Rheometer slideshow located on the C-drive. Starting at slide #139 there are many important settings for running this type of test.

There are numerous options in the procedure setup. Ensure that you have looked through all of them before you commit to run the experiment. To change the type of run you simply hover over the line of that step.



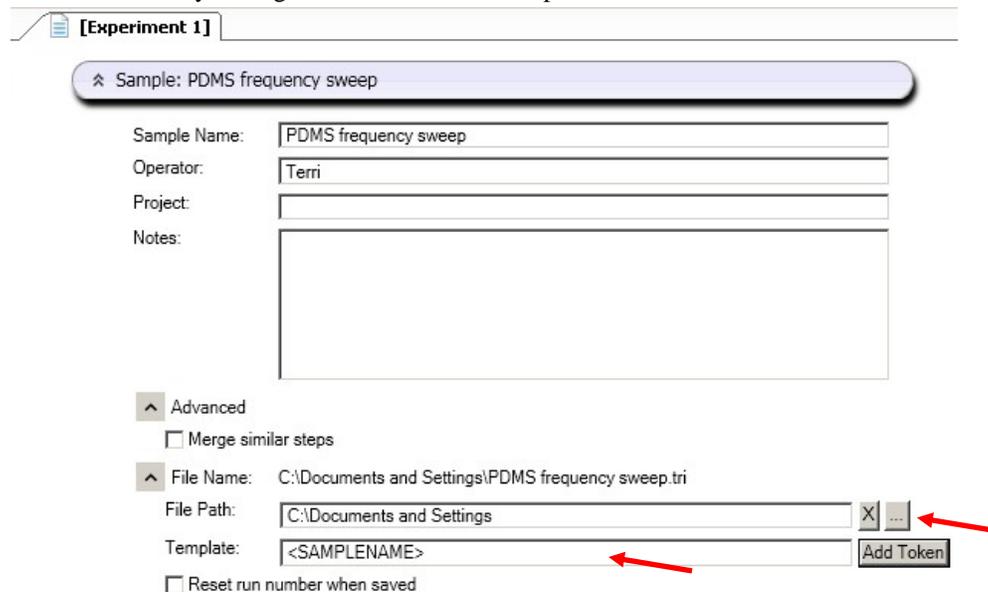
This is where new steps can be added, steps deleted, or steps can be rearranged. Make sure you look in all of the drop downs as well to make sure that there is nothing missed that is necessary.

16.4. Saving an Experimental Procedure

Saving a procedure that you know works well for a sample type is easy and ensures that there will be little variation between runs. Simply click the save icon on the blue procedure line to save it. Next to the save button is where previously created and saved procedures can be loaded.

17. Save an Experimental File

Select the directory/folder from File Path to save your data file. The name of the file can be the same as your sample name or others by adding more Tokens. The sample information can be entered in the Notes box.



18. Sample Prep

The amount of sample volume that is required, based on the dimensions entered in the Geometry step for a cone, parallel plate and concentric cylinder systems, can be found in the Geometry Information step.

When testing a dispersion or polymer melt using the Peltier or Parallel Plate ETC geometries, review steps 20 & 21.

When testing a polymer in pellet form, use the 25mm plates in conjunction with a melt ring, supplied in the ETC geometry kit, to contain the pellets. Place the melt ring around the lower 25mm stepped plate to form a well into which the pellets are placed, close the ETC, set a melt temperature, allow sufficient time for the pellets to melt. Lower the upper geometry until it is in touch with the polymer melt. Remove the ring, then follow Step 19 & 20.

To load disposable ETC geometry, first, attach the plates loosely to the upper shaft and lower stage, in order for the plates to slide without slipping out of the holder, and then lower the plates to within 5mm. Activate axial force control from the Control Panel with settings Axial Force: 5N, Sensitivity: 1N, Limit up/Down 100 μ m, Set initial value: On, Return to Window: On, Compression mode. Once the axial force has reached 5N, then tighten screws, this will ensure that the plates are parallel. Then raise the upper geometry and load the sample following step 18.

19. Gap Closure

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

- a. Manually enter the desired gap in the Control Panel – Gap window
- b. Automatically have the instrument go to the gap value entered in the Geometry file information.
- c. Manually raise or lower the gap by using the Front Control Panel arrows.

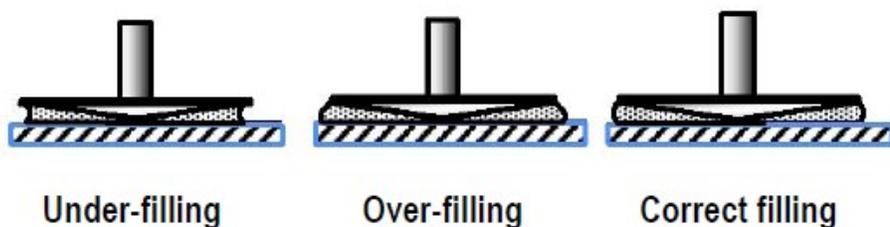
NOTE: The up and down arrows are also available in the Control Panel – Gap

NOTE: When using the cone geometry, the gap set must be equal to the truncation gap value that is scribed on the geometry shaft or stored in the smart swap file.

NOTE: When using the parallel plate geometry, the gap is variable, and should be between 500 microns and geometry diameter (microns)/10.

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

Load extra material and close the gap to a value of 5% larger than the required gap, so that excess material is expelled from between the upper geometry and lower plate, i.e. overfilled state. Then lock the bearing with the bearing lock button on the Front Panel in order to keep the geometry from rotating, and trim the excess material using a right edged tool. Then lower the gap to the final test gap. The correct filling condition is shown in the following picture.



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

21. Run a Test



Run test by selecting the start experiment icon .

22. Post Experiment Instructions

1. Ensure plates have cooled before touching them.
2. Remove dirty plates and clean if possible.
3. Shut ETC doors.
4. Set environmental test chamber to room temperature before leaving.
5. Leave geometry attached and instrument on unless directed by the lab manager.

Support and Service

Website: www.tainstruments.com

TA Instruments Applications Support: 302-427-4070 (M-F 8:00am-4:3pm EST)

TA Instruments Service Support: 302-427-4050 (M-F 8:00am-4:3pm EST)