MATERIALS CHARACTERIZATION FACILITY
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DHR-30 S.O.P

Instrument:

Discovery Hybrid Rheometer (DHR-30)

Software:

Trios

Purpose:

To discover the viscosity and viscoelasticity of a material

Acknowledgements:

This SOP pulls from the TA-instruments user manual for the DHR-30 and is heavily based on the "Intro Guide to DHR" from the Cornell Center for Materials Research

To Begin:

- **Know the specifications of the instrument.**

Combined Motor and Transducer (Single Head)

Bearing Type-Thrust	Magnetic
Bearing Type-Radial	Porous Carbon
Motor Design	Drag Cup
Minimum Torque Oscillation	0.3 nN.m
Minimum Torque Steady Shear	1 nN.m
Maximum Torque	200 nN.m
Torque Resolution	0.05 nN.m
Minimum Frequency	1.0E-7 Hz
Max Frequency	100 Hz
Min Angular Velocity	0 rad/s
Max Angular Velocity	300 rad/s
Displacement Transducer	Optical Encoder
Displacement Resolution	2nrad
Steo Time, Strain	15 ms
Step Time, Rate	5 ms
Max Normal Force	50 N
Normal Force Sensitivity	0.005 N
Normal Force Resolution	0.5 mN
Peltier Plate	-40 - 200 C
Environmental Control System	-160 – 600 C

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1. **Setup**

- Log on to the computer. The password is: Rheometer

- Turn on the controller for the rheometer. The power switch is on the back of the control box

Control Box for the Rheometer

  - **Make sure compressed air is flowing to the rheometer.** This rheometer is supplied by house compressed air, but make sure that the proper pressure is being supplied **before** turning the rheometer on.

  - Failure to do so will result in damage to the radial air bearing, incurring large charges to your lab for repairs $$$

  - Pressure requirements for all DHR models: 30 psi

- **Remove the black bearing cap** by holding it in place while turning the draw rod knob at the top in a counter-clockwise direction. Once the bearing cap is removed, make sure that the spindle rotates freely. If the bearing cap is not present, ignore this step.

  - NOTE: Turning on the rheometer while the bearing cap is on the rheometer will result in damage to the radial air bearing, incurring large charges to your lab for repairs $$$

- Turn on the Power to Rheometer using the power button. 

  - When using a Peltier control device, please ensure that the coolant supply is turned on. If not using the Peltier control device, make sure the coolant supply is turned off before swapping stages.

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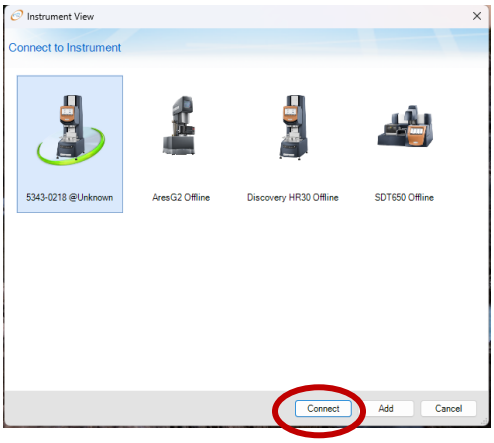
- When the instrument has finished the system check, start the instrument control software TRIOS  . Then click "connect" with the Rheometer selected to begin communication to your rheometer.

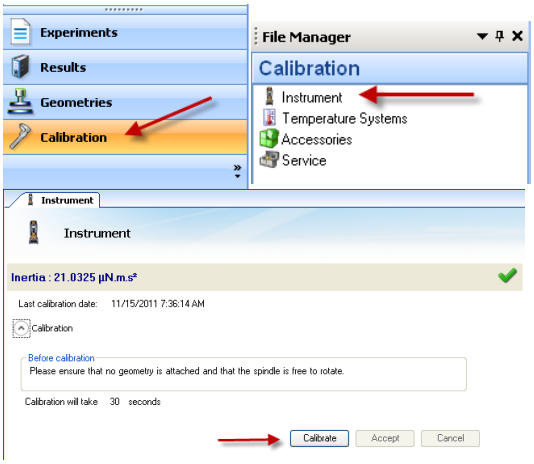


- Choose the Control Panel icon from the Home ribbon, 

, 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.

2. Calibration and Inertia Checks

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



  - The value for the instrument should not change by more than 10% of the original Inertia value. If you see it fall outside this range, contact facility staff to check on the instrument so that you do not incur repair charges.

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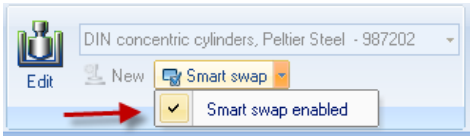
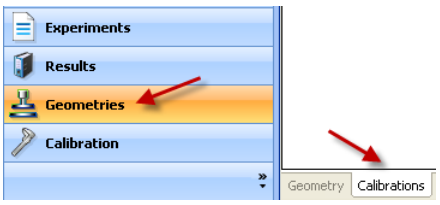
3. Select a Geometry

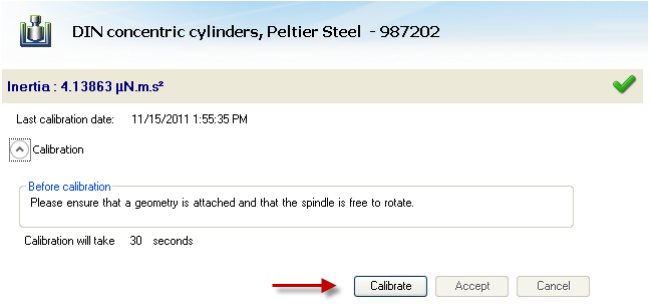
- Lock the radial bearing using the lock button  on the rheometer. Attach test geometry by sliding it up the drive shaft and hold it stationary while turning the draw rod at the top in a clockwise direction. If the smart swap geometry option "smart swap enabled" is selected, the appropriate geometry file is automatically applied to your experiment. If the smart swap geometry option is disabled, choose the appropriate geometry from the list of geometry files previously created.



- 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 the *Geometries Tab, Calibrations page* and then *Calibrate*.





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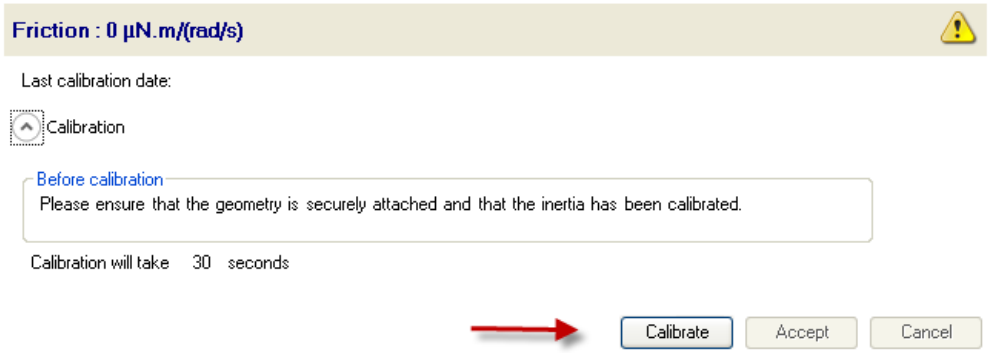
- **Bearing Friction Correction:** A magnetic bearing is used to set the drive shaft afloat and provide virtually friction free application of torque to the sample. However, there will always be some residual friction. To overcome this, the software has a bearing friction correction that should be activated. Bearing friction correction can be found just below the Geometry Inertia calibration.

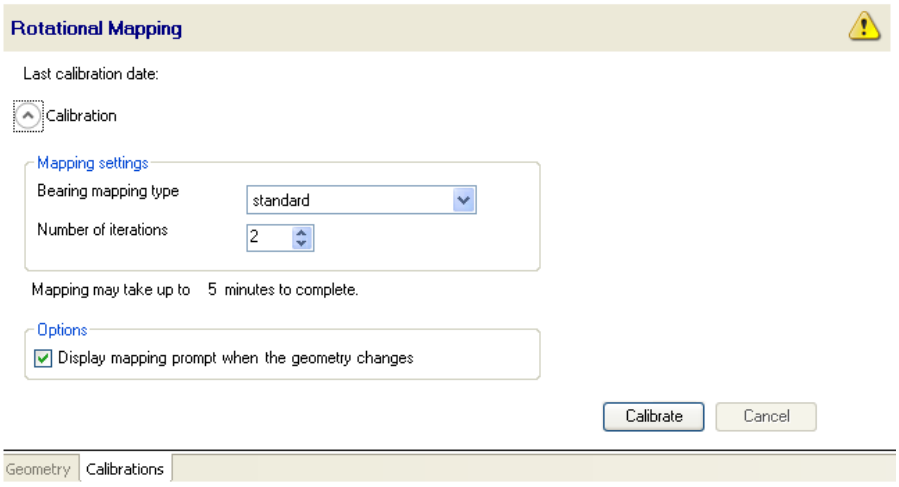


  - NOTE: Please ensure that the Instrument Inertia and Geometry Inertia have been calibrated before determining the bearing friction correction value.

- **Mapping**

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

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



  - There are three levels of rotational mapping – fast, standard, and precision. It is also possible to perform multiple mappings iterations.

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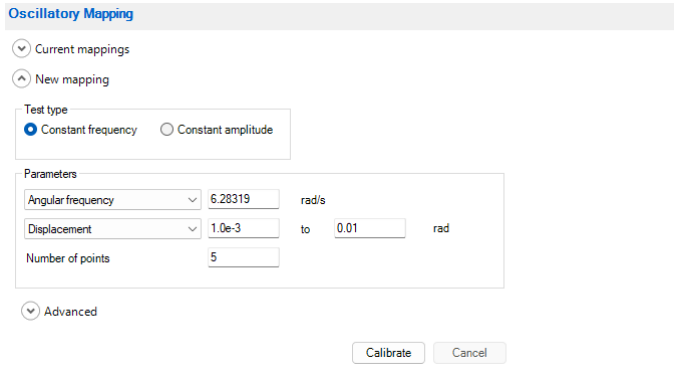
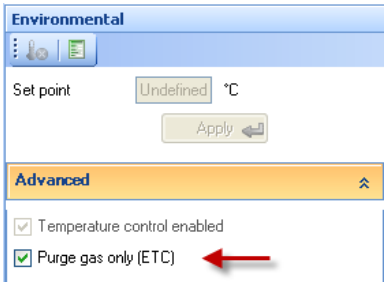
- Oscillation Mapping: This mapping will perform a baseline subtraction only when using the continuous controlled strain mode and will improve the performance for low torque, low displacement data.
  - If running this mode, find the mapping prompt under the calibration page, where instrument inertia was calibrated. Perform this mapping without a geometry attached.



4. Select a Temperature System

➢ Attach the appropriate Smart Swap lower geometry temperature stage/system. To do this, unplug the lower geometry , press the smart swap button  , remove the lower geometry, place the new lower geometry on the rheometer, then plug the circular connector into the rheometer with the red dot facing up.

- NOTE: If the rheometer is equipped with an ETC and it is being used for testing, set the ETC flow meter to about 10 L/min.
- 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.



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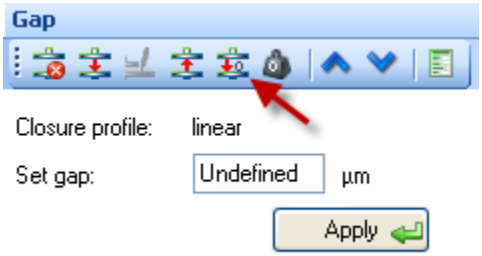
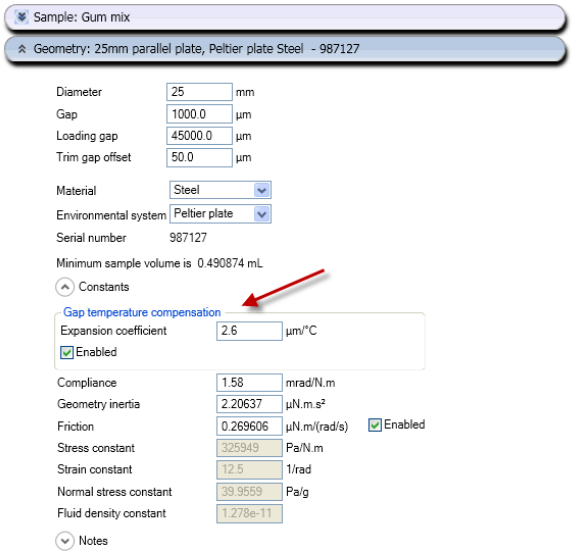
5. Zero the Geometry Gap

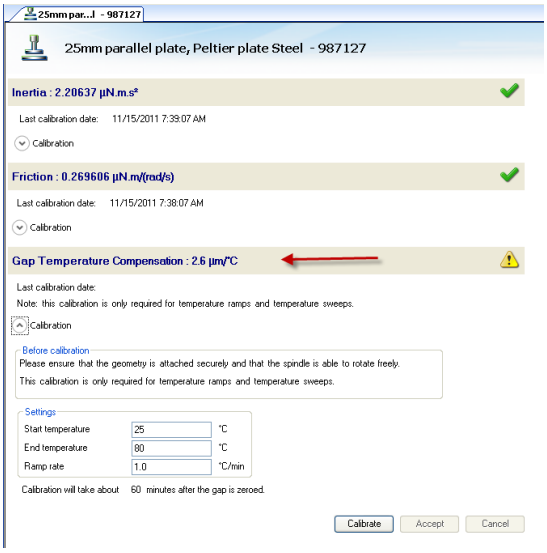
- Choose the zero gap icon located under GAP from the Control Panel and follow the directions on the screen.



  - 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.

- **Gap Compensation**
  - NOTE: Predetermined values can be entered into the TRIOS software. The gap compensation check box must be active, which is located within the Geometry Constants section of the test procedure. Gap Compensation needs only to be used when testing over a temperature range. If controlling normal force throughout an experiment, the gap compensation value should be activated.





  - To correct for changes in gap due to temperature use the gap compensation wizard located in the Geometries Tab, then Calibrations page for your chosen test fixture.

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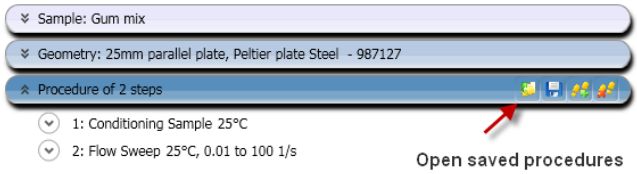
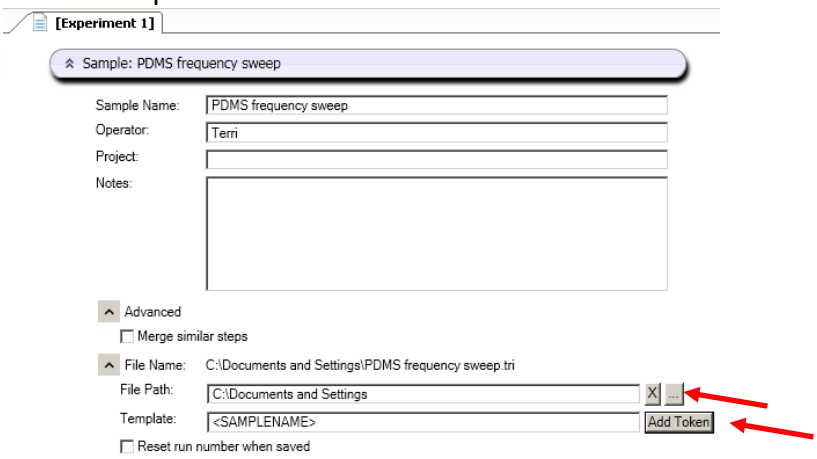
6. Set up a Procedure

- 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.

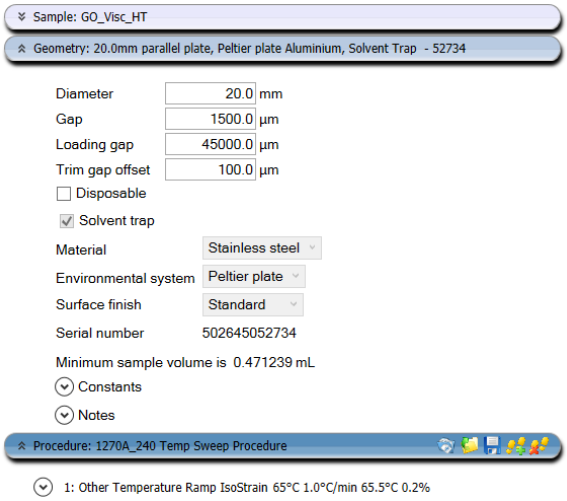


- **Save an Experimental File**
  - Select the directory/folder from File Path to save your data file. The name of the file can be the same as your sample name or others by adding more Tokens. The sample information can be entered in the Notes box.



7. Sample Loading

- 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.



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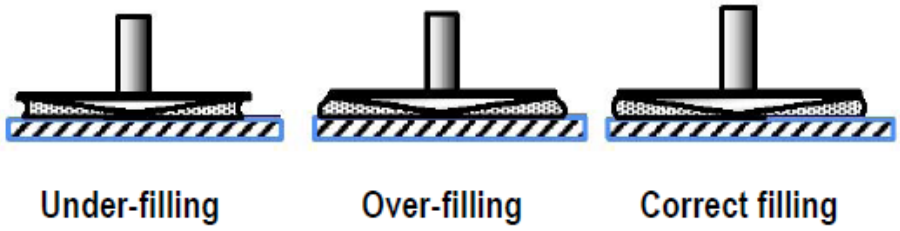
8. Gap Closure

- After loading a sample, the gap is closed by three different methods:
  - Manually enter the desired gap in the Control Panel – Gap window
  - Automatically have the instrument go to the gap value entered in the Geometry file information.
  - 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. 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. When using the parallel plate geometry, the gap is variable and should be between 500 microns and geometry diameter (microns)/10.

9. **Trimming the Gap** (cone or plate geometry systems)

- Load extra material and select "go to trim 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

10. Run a Test

- Run test by selecting the start experiment icon 

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  - Your data will be displayed in real time in the results tab. When all the data is collected, trios can analyze your data using the analysis tab, or you can right-click a graphed data set and select an analysis from the dropdown menu. Export data in .csv formats by right-clicking them from the left panel and select export. Data can also be exported from the file dropdown menu in the upper left corner of TRIOS. Collect your data from the computer using a thumb drive.

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11. Clean up

- When finished with running your experiment, lock the geometry and raise it from your sample. Remove the geometry and clean it off using a non-corrosive solvent, kimwipes, and brushes.

- **Do not use abrasive brushes directly on the Peltier plate.**

- Return the geometries you used to their respective boxes, close TRIOS, and **turn off the DHR-30** using the power button  .

- If the black bearing cap is present, place it back onto the bearing.

- The control box for the DHR-30 can be left on between sessions.

Notes on Setting up the Torsion Rectangular system

- *Use the provided credit card slice instead of a steel shim for alignment*

- Follow prompts from the front screen of the DHR-30 when attaching the Torsion Rectangular system for calibration and alignment.

- Make sure to zero the axial force via the control panel when nothing is attached. Then zero the gap.

    - NOTE: Manually align the upper and lower clamp faces, and then lock the bearing from the Bearing Lock button on the Front Control Panel. This will electronically inhibit any rotation of the geometry and allow one to zero the gap without the geometry rotating.

- After the gap is zeroed and you've loaded your sample at the desired height, you can either condition your sample via a conditioning step in the experimental set up, or you can manually condition your sample or apply a pretension using Axial Force tab under the control panel to apply a specific force before running your experiment.

• TA Instruments rheology applications support line
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