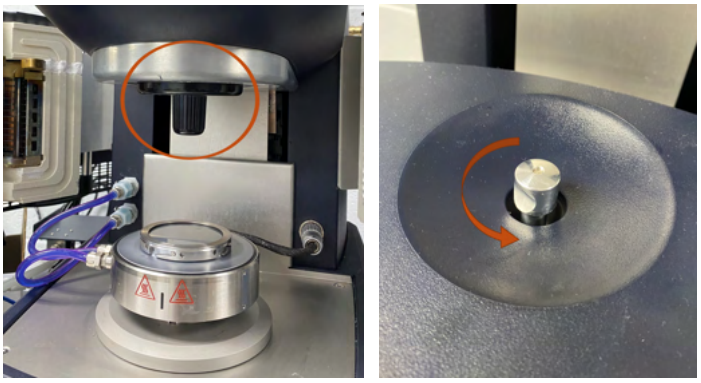
TA Hybrid Rheometer (RHEO2)
Quick Start Guide

Before Starting

1. Remove the bearing clamp. Hold it securely and turn the draw rod on top of the instrument head counterclockwise to release it



(a) Bearing Clamp (b) Draw Rod

Figure 1: Motor security clamp

2. Turn ON the RHEO from the front button and allow it to initialize

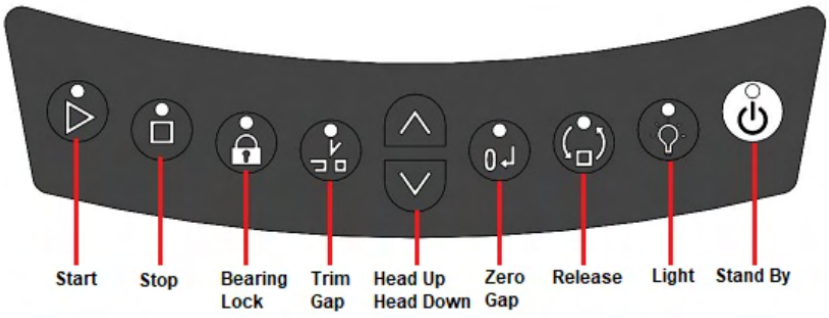


Figure 2: Instrument front panel with buttons labeled

3. Depending if temperature is required
    - **No temperature required.** It is recommended to use the Peltier plate because the geometries are shorter, which reduces the risk of the shaft becoming unstable
    - **Temperature required.**[1] If solid, rigid materials are used ETC is recommended[2]. If liquids, gels, or highly fluid materials are used Peltier Plate is recommended
4. If temperature is controlled by the Peltier Plate, turn ON fluid circulation from the Thermo CUBE cooling system

Figure 3: Thermo CUBE cooling system. Placed bellow the instrument bench

Configure Measurement

1. Launch TRIOS software

[1]For additional details, consult **Appendix b. Setting Measurements with Temperature**

[2]This option is intended for performing Dynamic Mechanical Analysis (DMA); however, we currently don't have the necessary accessories in this DEMO version of the instrument. In the laboratory, the *RSA-G2 Solid Analyzer*

Figure 4: TRIOS deskpot icon

2. **Instrument View** window will display. Select **5343-0385 / Discovery HR20** instrument and click **Connect**. The main page will display

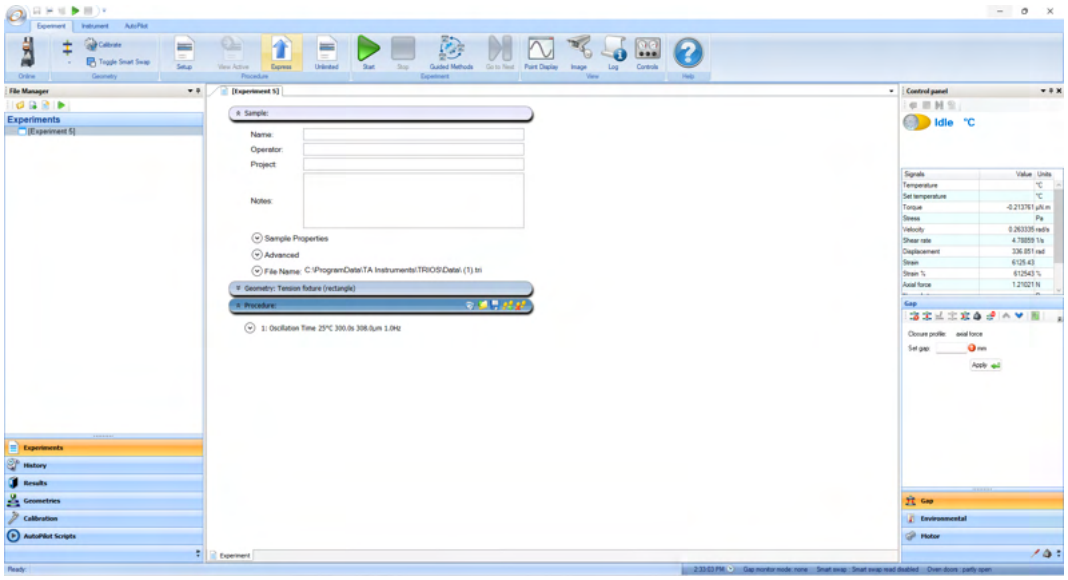


Figure 5: TRIOS main page

3. Install the desired geometry[3]. Place it into the bearing, aligning the 0° angle mark with the instrument head mark. Rotate the draw rod on top clockwise to install the geometry[4]

[3]For additional details, consult **Appendix a. Adding the Measuring System**
[4]It should be screwed finger tight, but not forced

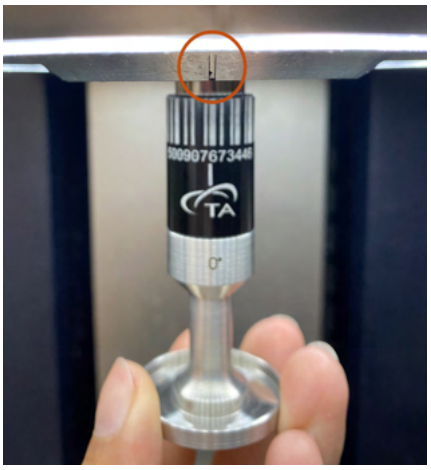


Figure 6: Alignment of geometry with instrument head

4. If the geometry has the Smart Swap system, it should rotate a few seconds and will be automatically identified by the RHEO. If not, go to the **Geometries** section located at the bottom left of the screen and introduce the current geometry

5. Go to **Gap** section located at the bottom right of the screen. Click the **Zero Gap** symbol. The automatic gap zero position routine should start[5]

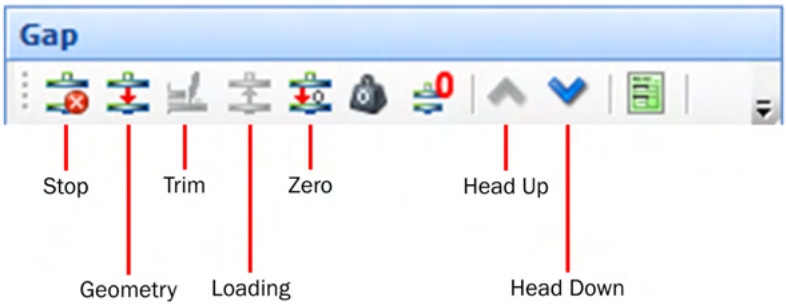


Figure 7: Gap section in software

6. Go to the **Calibrate** section located at the menu bar and calibrate the **Intertia**, **Friction** and **Rotational Mapping**. If temperature ramps are required, also calibrate the **Gap Temperature Compensation**

7. In the **Gap** section adjust the plates gap before the measurement

[5]The oven doors should be fully opened for the head go up after setting the zero gap. Current status can be checked at the bottom right of the screen

- Click the **Loading** symbol to move the instrument head up[6]. Place your sample in the lower plate[7]
- Click the **Trim** symbol to place the geometry close to the sample
- In the front panel click the **Lock** button to prevent movement of the geometry. Remove away any excess material an unlock the geometry by pressing the **Lock** button again
- Click the **Geometry** symbol to move the geometry to the value specified for the current geometry

8. Allow samples to relax before beginning testing by monitoring the normal force in the user interface and waiting for it to decay to close to zero

9. In the **Experiment** section of the main page, identify your sample and configure the procedure[8]

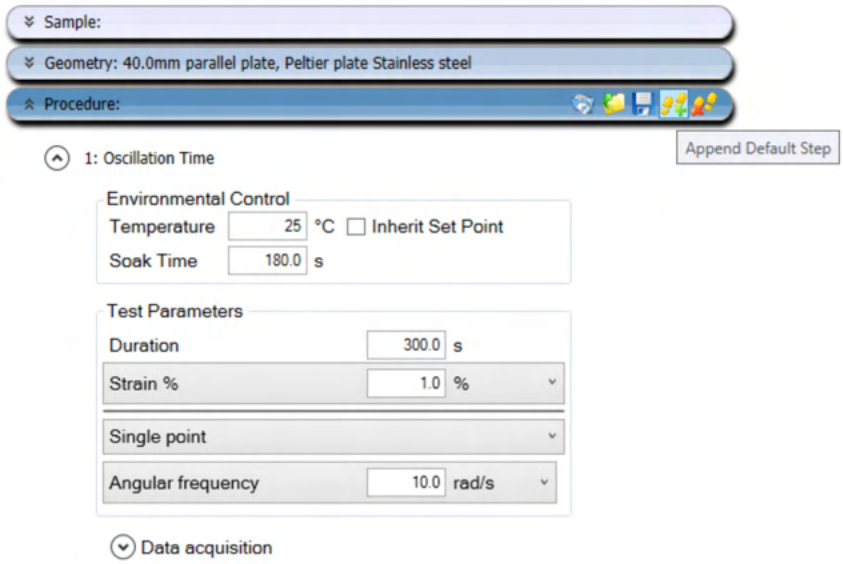


Figure 8: Procedure configuration

10. If more than one test needs to be performed, click the **Append Default Step** symbol. Another subsection will display[9]

[6] If more space is needed, click the **Head Up** button to raise the instrument head further
[7] For additional details, consult **Appendix a. Adding Measuring System**
[8] For additional details, consult **Appendix c. Building a Procedure**
[9] A delay between tests is recommended, so the sample structure can reconstruct after the shear applied. Set a **Soak Time** in continuous procedures.

11. Once your measurement is completed

    - Go to the **Gap** section and click the **Loading** symbol to release the geometry from the sample
    - Remove the geometry in the inverse way as you placed it
    - Clean the lower plate and the geometry using light duty tissue wipers and water. To eliminate any excess material, use isopropyl alcohol
    - Store the geometry
    - Place back the bearing clamp back in the instrument head in the inverse way as you removed it

12. To save your data click on the TA logo in the left up corner of the screen. In the drop-down menu click **Export** and select the method that best suits you to analyze your data later.

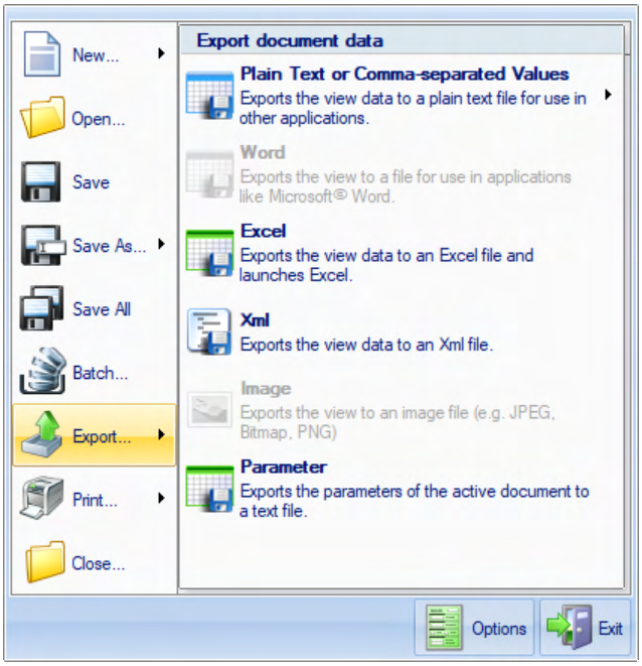


Figure 9: Data exportation

13. If no user is signed up in CoreResearch to use the RHEO2 on the same day or early the next day, turn OFF the instrument from the front panel

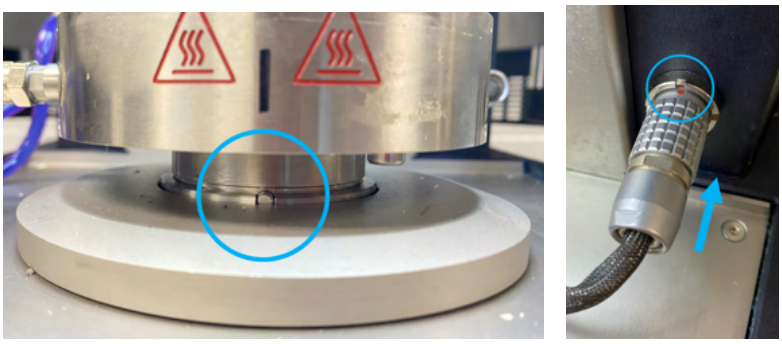
Appendix

a. Adding the Measuring System and Sample Loading

1. Install Lower Plate

The laboratory has both the Peltier Plate and ETC bases available. To place them on the rheometer a similar procedure should be followed.

Fig. 10 shows the main considerations to keep in mind when placing the Peltier Plate or ETC base on the instrument.



(a) Base fitting of the environment system plate with the instrument base

(b) Smart Swap Plug

Figure 10: Considerations to place lower plate into instrument button stage

Before placing any base on the instrument stage, temporally release the magnet by pressing the Release button on the instrument's front panel.

In **Fig. 10 (a)**, it is shown how the base should be fitted into the instrument button stage. The groove on the instrument base must align with the slot on the Peltier/ETC base. When finished, press the Release button again to fully attach the base to the instrument. In **Fig. 10 (b)**, the cable connecting the base to the instrument for temperature control is shown. It should be connected so that the red dot is facing up, as illustrated. **********para sacarla no me acuerdo si la parte de atras se tira para atras o adelante.... perguntar renata

2. Install Measuring System (MS)

To install the geometry in the instrument head, place it in the bearing and align the 0° angle mark as shown in **Fig. 6** . To secure it, rotate the draw

rod clockwise. Some geometries are automatically recognized by the instrument's Smart Swap system. If this is the case, the geometry will begin to rotate, and its status can be checked in the **Geometry** section, where a ticket will appear over the corresponding geometry logo. If the geometry is not automatically recognized, in the **Geometry** section can be selected.

Before placing the sample on the lower plate and performing the desired measurements, zero gap and calibrations should be configured, as indicated in the **Steps 5** and **6** of **Configure Measurement**.

3. Sample Loading

For accurate sample placement, ensure the sample is as centered as possible between the mounting surfaces. Off-center loading may misalign the motor shaft and affect data accuracy. **Fig. 11** shows a properly loaded sample with correct alignment, trimming and geometry gap. The goal is to create a slight convex angle at the sample's edge after removing excess material and setting the Geometry Gap. This can be seen by comparing the second and third images in **Fig. 11**.

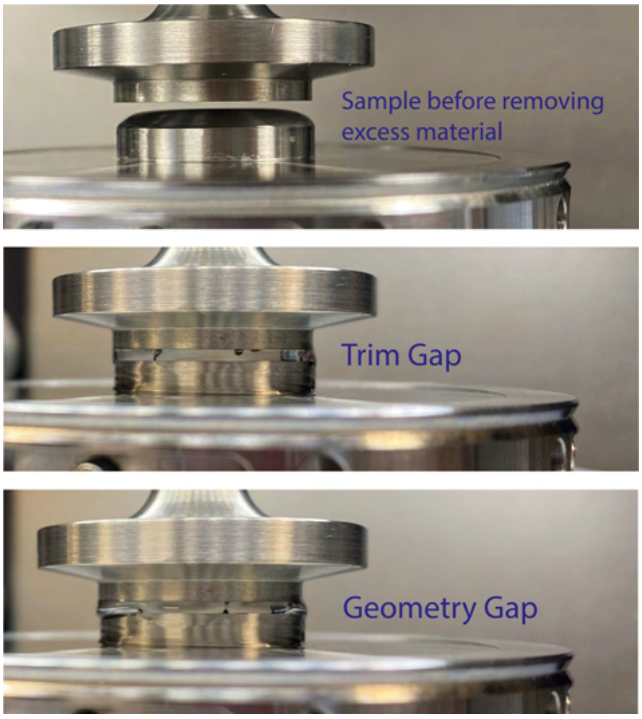


Figure 11: Proper loading of sample for a PP MS.

Fig. 12 exemplifies the method for cleaning excess sample during trimming.

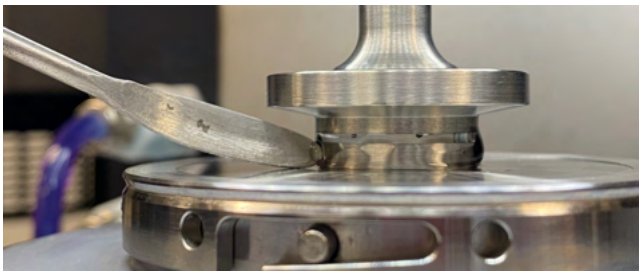


Figure 12: Sample during trimming for a PP MS.

b. Setting Measurements with Temperature

If planning to use temperature changes in the measurements, it's important to calibrate the **Gap Temperature Compensation**. This calibration considers the volume changes in the geometry due to its thermal expansion. Therefore, it will record the volume change during a temperature ramp and it may take 1 or 2 hours to complete. For this reason, it is recommended to **contact the trainer in charge Renata Garces** (email: *renata.garces@duke.edu*), to set the calibration with the desired MS before your scheduled time for using the rheometer.

For measurements with temperature control, an environment system should be picked. Currently, the **Peltier Plate** and **ETC** are available. **Fig. 13** shows both control systems.

(a) Peltier Plate

(b) Environmental Test Chamber (ETC)

Figure 13: Environmental Control Systems

The use of any of these environmental systems require to install the corresponding base into the instrument bottom stage. The instructions can be found in **Appendix a.1. Install Lower Plate**.

- **Peltier Plate.** It uses a peltier thermoelectric effect to control the temperature accurately, with rapid heating and cooling, that goes from -40

°C to 200 °C. This system requires a cooling system connected to the instrument. Instructions in **Step 4** of **Before Starting**. The use of this control system is recommended for samples that does not require compression, stretching or torsion measurements.

- **ETC.** It employs a combination of radiant and convective heating and has a temperature range of -160 °C to 600 °C with heating rates up to 60 °C/min. This chamber enables the characterization of solid samples in torsion by enclosing the measuring system inside an oven. The **Status Bar** in software reports the current state: open, partly open, or closed. Temperature control is only active when the doors are closed; also, the instrument head will move only if the doors al fully opened.

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 configure in the **Environmental** panel of the software. The options vary based on control system installed.

c. Building a Procedure

This section presents some of the principal procedures available to perform in the TA Discovery HB 20 and a brief description and purpose of each configuration.

1. General Configuration

Previews Recommendations

- If materials expand, contract or slip during testing it is recommended to change the geometry gap during measurement. Use the **Axial Force**[10] function to ensure that contact is maintained between the geometry and sample.

Environmental Control

Temperature selection to maintain throughout the test.

Inherit Set Point maintains the previously specified temperature at the start of this step.

[10] Can be enabled/disabled and set in the **Axial Force** section of the **Instrument Control** panel. Activate it after loading the sample, but before enabling temperature control.

Soak Time is the amount of time to delay data acquisition at the start of the step, typically to allow for temperature equilibration[11].

Wait for Temperature allows you to wait until the measured sample temperature reaches the specified temperature before beginning data acquisition.

2. Oscillation Configuration

This procedures calculate the storage modulus (G'), loss modulus (G''), and the complex viscosity ($\eta*$), as functions of the testing parameter.

Frequency Sweep Test. The frequency is varied linearly or logarithmically. The strain, stress or torque amplitude of the sinusoidal deformation remains constant (and also the measuring temperature). Strain, stress and phase are recorded.

This configuration is used to investigate time-dependent deformation behavior. Short-term behavior is simulated by rapid motion, i.e. at high frequencies, and long-term behavior by slow motion, i.e. at low frequencies.

Temperature Ramp. The temperature is varied continuously at a desired rate of °C/min. Both the frequency and the amplitude are kept at a constant value in each test interval. Strain, stress amplitude, and phase shift are recorded.

Provides information of how a material changes with temperature. The most important application is the measurement of thermal transition in materials or to simulate cure cycles[12] of thermosetting materials.

Temperature Sweep. The temperature is varied in steps. The oscillation measurements are performed under isothermal conditions. In contrast to temperature ramps, where frequency is constant, the isothermal condition allows for multiple frequencies. The frequency can be varied linearly, logarithmically, or in discrete steps. Strain, stress amplitude, and phase shift are recorded.

The primary application of a temperature sweep is frequency-temperature

[11] Usually 3 min soak time is enough when the temperature change is not too large.

[12] The material transitions from a liquid or semi-solid to a solid and rigid state when exposed to heat. During this process, chemical reactions form cross-links between molecules, transforming the material from a 2D to a 3D structure

superposition, which helps extend the experimental frequency range. By performing this test, we can evaluate how temperature influences physical properties, such as phase transitions and structural changes, all while maintaining constant dynamic-mechanical low-shear conditions within the linear viscoelastic (LVE) range.

Time Sweep. Consist of a constant oscillation performed during a desired amount of time. Temperature, frequency, and strain or stress amplitude are 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 thermal/mechanical/etc stability or the evolution of materials with time at constant temperature.

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 non-linear viscoelastic behavior. In this region, the material response is not sinusoidal anymore, and odd higher harmonics become important. The onset point of non-linear behavior in a strain sweep of structured fluids is often used to determine the yield behavior.

3. Rotational (Flow) Procedure Configuration

Temperature Ramp. Temperature is varied continuously and the instantaneous viscosity is recorded. Torque, Stress, Velocity or Shear rate can be configure as constant during measurement.

Equilibrium flow must be reached first before ramping the temperature, therefore, it is better to start at an elevated temperature, and ramp the temperature from high to low. The ramp rate must be slow enough to reduce temperature gradients in the sample to a minimum.

This is a fast test method to determine the temperature dependence of a material's viscosity.
Continuous Ramp. A rate or stress ramp can me performed. The rate or stress is varied linearly or logarithmically up and down, while the uncontrolled parameter is recorded over time; e. g. in a stress ramp, the shear rate is recorded.

The rate ramp was originally designed to evaluate the time-dependence with shear rate for structured fluid and detect thixotropic behavior. The ramping up, combined with the ramping down, of the shear rate is also referred to as a thixotropic loop test.

The main application of the stress ramp experiment is to evaluate the yield stress of structured fluids.

Sweep. The rate or stress is varied linearly or logarithmically, while the uncontrolled parameter is recorded in the steady state (equilibrium). The averaged equilibrium stress, or rate and viscosity, are recorded as a function of the strain rate or stress. Opposite of the rate ramp, the step rate experiment provides equilibrium data, and the results are not dependent on the test time.

The rate sweep experiment is the method of choice to determine the zero shear viscosity of materials with long relaxation times.