SOP: Interfacial Hybrid Rheometer

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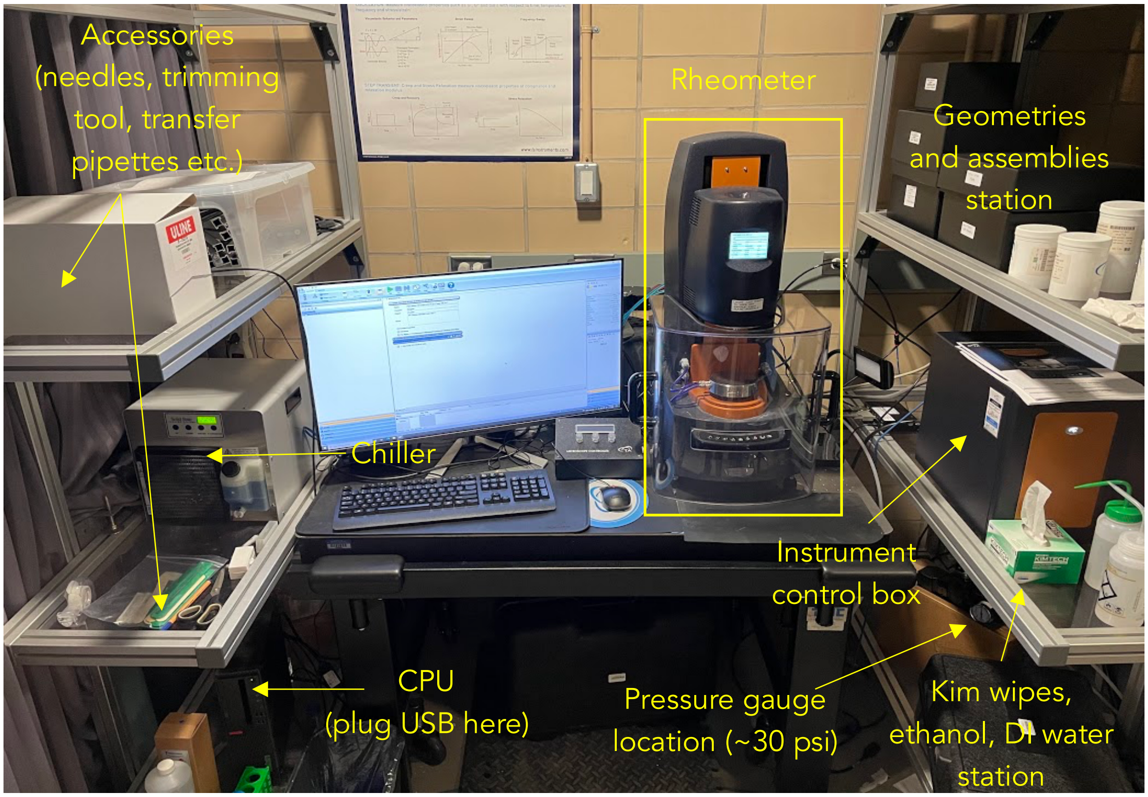
Equipment Overview

The DHR-3 rheometer from TA instruments is used for measuring rheology of fluids and viscoelastic solids (semi-solids). It works by relating material properties to see how they respond to an applied stimulus (mechanical or thermal stress or strain).

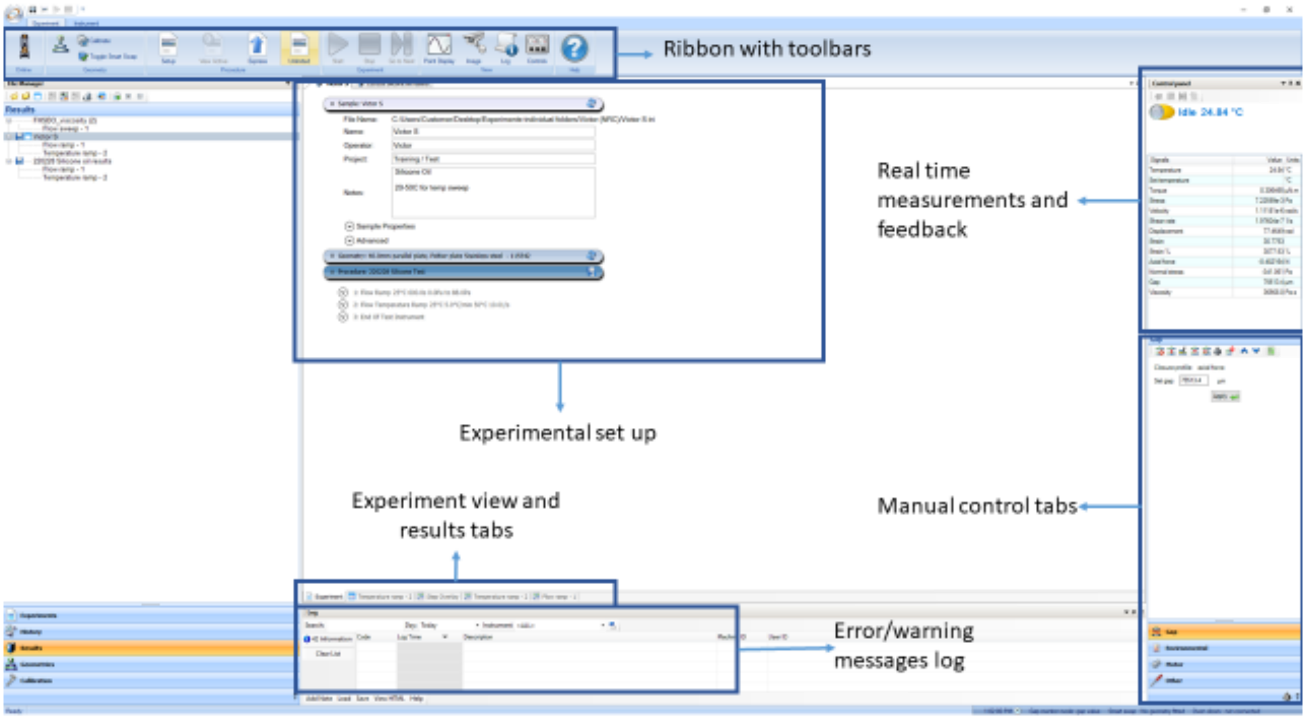
Notes before use:

- Ensure instrument is ON before starting the TriOS software
- While some literature in the help guide permits use of solids, this particular instrument does not have the mechanical analyser, hence use only fluids
- Users should know the melting point of their sample, this is important for the cleanup process as it will be difficult if the sample solidifies
- Do not use any corrosive chemicals that can damage the geometry and bottom assemblies
- Wipe down the surfaces and geometries before and after the experiment
- The rubber mats and floor mats are to protect the geometries in case of an accidental drop
- Please notify staff in case the chiller sounds an alarm
- Operate within the operational limits of the instrument (see table on page 7)
- No chemical powders (ie. graphite, carbon nano tubes) or sample preparation should be performed at the rheometer station



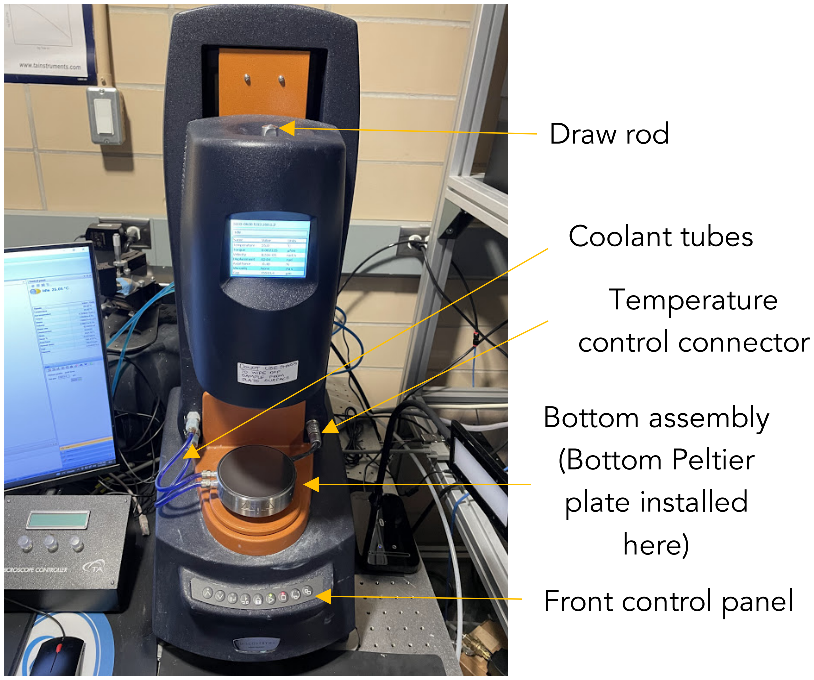
Instrument setup

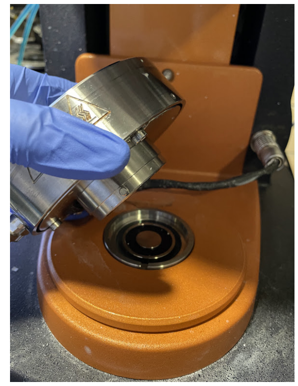
1. Login to the Rheometer from LMACS
2. Check to ensure that the air pressure to the instrument is 30 psi (±3 psi) and verify that the instrument status shows 'idle'. If the instrument is OFF follow these steps in the stated sequence.
    a. Turn ON the air pressure to ensure it is 30 psi (±3 psi)
    b. Remove the protective cap from the rheometer head (where the geometry connects)
    c. Turn ON the instrument using the switch at the back of the instrument control box (red CPU- right of the instrument)
    d. Turn ON the computer (slim black CPU on the left bottom shelf)
3. For an understanding and background on theory of rheology in context with this DH3 Rheometer, review the resource titled 'DHR_Rheology_Theory' located on the desktop (Copy or share it to review it later).
4. Open the TriOS software and connect to the DHR3 instrument. (TriOS is a free software available from TA instruments. Consider downloading on your personal/lab computers for post measurement analysis of data. (https://www.tainstruments.com/support/software-downloads-support/downloads/)



5. Determine the test geometry and the base set-up for the required test. CRAFT offers a 40 and 60 mm flat plate, a 60 mm sand blasted plate, a 1 deg cone plate, a bi-cone plate for interfacial rheology, and a concentric cylinder geometry.

   As for the bottom accessory, CRAFT offers a bottom and top Peltier plate assembly, a Peltier concentric cylinder – all three are equipped with Smart Swap technology (automatic detection), and a cup for interfacial rheology measurement support. An environmental chamber is also available for samples requiring controlled environment.
   (This SOP is prepared with a bottom peltier plate assembly in focus)

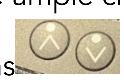
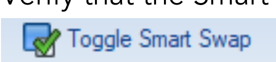


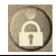


Magnet ring slot

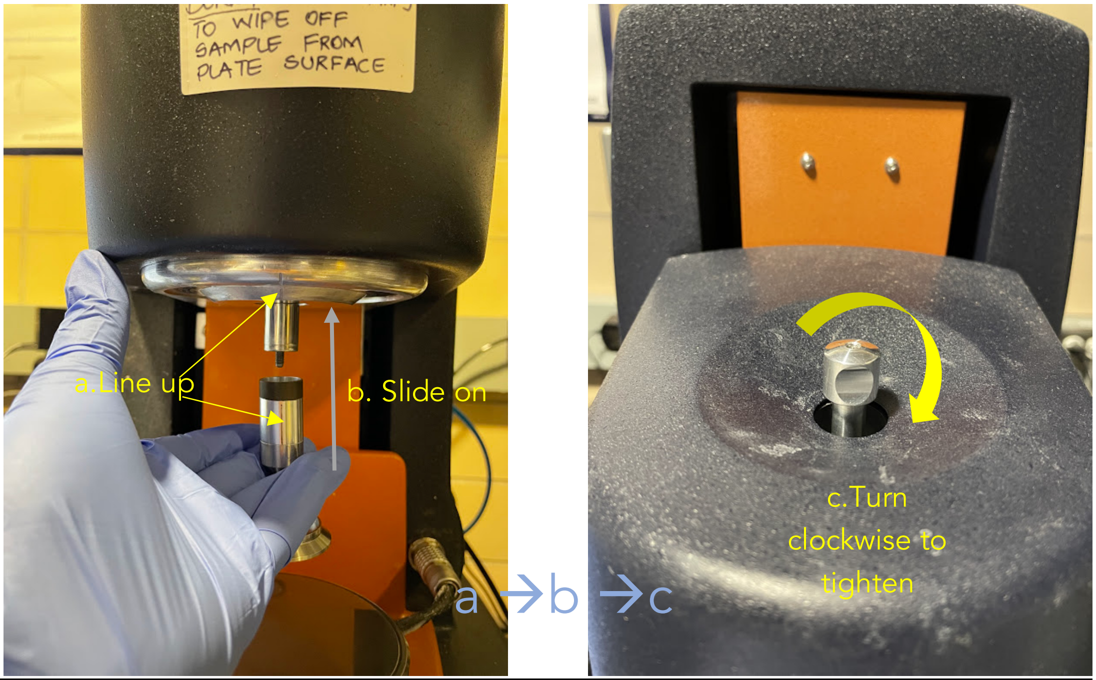
6. To install the bottom Peltier plate, press the magnetic swap button  on the front panel twice (should be a solid light). Place the plate in the ring slot, ensuring the pin lines up with its slot and press the swap button again to enable the magnet (the light should disappear). Connect the coolant recirculation tubes to the connections and the temperature control connector to the connector on the instrument. To verify detection check the TriOS software, under environment tab- look for the status 'peltier plate: idle').
7. The chiller is initially in STANDBY mode. Activate the chiller system by pressing the START/STOP button on the front panel of the chiller. Set the temperature at least 10 degree below the lowest test temperature (recommendation). If the chiller is powered OFF, power it up using the switch at the back of the chiller.

Note: the temperature range of the chiller is -5°C to 200°C

8. Before connecting the top geometry,
    a. Ensure ample clearance by driving the head manually using the travel control buttons  on the front panel.
    b. Verify that the Smart Swap is enabled (either check mark on 'toggle Smart Swap' 

in the top ribbon, or status at the right bottom edge).
    c. Wipe down the geometry with ethanol and water to ensure no debris on the surface or in the inside groove.
9. Lock the motor by <u>holding down the lock button</u>  <u>on the front panel until an audible beep is heard</u>. The light on the button should be flashing at this stage.

10. Align the vertical line (co-axial to the geometry) on the geometry with the line on the rheometer head. Slide the geometry onto the motor shaft till it tops out (cannot slide any further), keeping the geometry vertical throughout this installation (don't tilt the geometry). Rotate the draw rod on the top of the head in clockwise direction until the attached geometry just feels the turn (just tight enough but not too tight, do not fight the motor when tightening). The geometry should start spinning the instant the draw rod is tightened and then stops after a few seconds. When this happens, let go.

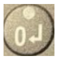


11. In TriOS , Click **Calibrate**, in the top ribbon, towards calibrating and verifying a proper installation of the geometry.
    a. Perform the inertia calibration, friction calibration and rotational calibration- in that order (recommendation).
        i. The inertia values are acceptable if they differ by 0.1 or less. If not, re-attach the geometry again.
    b. Step away from the set up during this calibration process and do not touch the table.
    c. Ensure that the calibrated values are under $\pm$0.1 units of the last calibration.
    d. If the values are significantly different, uninstall and reinstall the geometry.
    e. If the values continue to be different, please notify the staff/ trainer.

12. Consider the Gap temperature compensation calibration only if the planned experiment involves testing the sample at multiple temperatures. If the planned

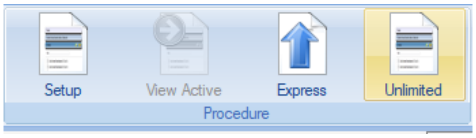
experiment is to be carried out at a fixed temperature, set the temperature in the environmental tab and then proceed to calibrate the zero gap on the instrument.

Experiment setup

13. Setup the temperature in the Environment tab, and click **Apply**.
14. Zero gap measurement identifies the vertical axis value at which the geometry makes contact with the bottom Peltier plate. Press the zero gap button  on the front panel or in the gap tab in TriOS. (The gap temperature compensation calibration process effectively calibrates zero gap across a range of defined test temperatures incorporating any expansion of the geometry and Peltier plate with temperature).

Note: For particles, the gap is generally set to 10x it's size. For example, a 20 um particle should have a gap size of 200 um.

15. Define a new experiment or import the experiment you plan to rerun or build up on. <u>Name the experiment and operator and define the file name and file path in the sample tab</u>.
16. In the geometry tab, define the Gap and trim gap (refer to literature or file titled: 'DHR_Rheology_Theory').
    a. Gap is the test gap or the sample thickness you intend to perform measurements on
    b. The recommended trim gap is 5% of the gap.
17. Note down the recommended minimum sample volume in this geometry tab.
18. Set up the experiment by using literature or file titled 'DHR_Rheology_Theory' or the poster on the wall towards identifying the appropriate test conditions. Some test conditions may not be available in the express set up mode (which is the default mode). Switch to unlimited mode, to access more test and control parameters (Optional: for me, I'd put this step when checking with the toggle switch at the beginning).



Testing regimes available each test condition are as follows,

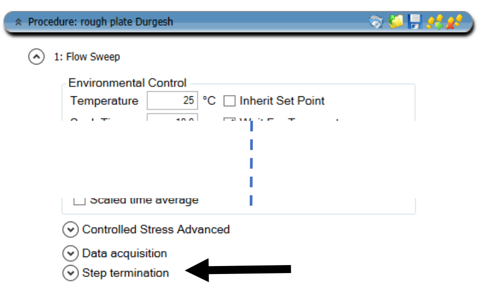
Flow	Oscillation	Step (transient)
• Temperature Ramp • Ramp • Sweep • Peak Hold	• Frequency • Temperature Ramp • Temperature sweep • Time • Amplitude	• Creep • Stress relation • Stress growth • Repeated creep

	• Fast sampling • manual	

While setting up the experiment, remember that the instrument only has finite capabilities. A few operational limits to consider before starting the experiment,

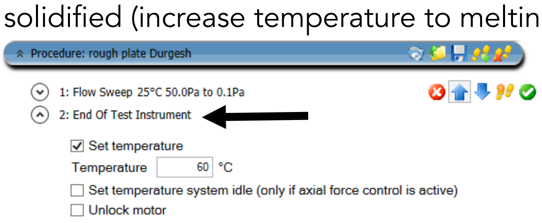
Minimum Torque (nN.m) Oscillation	0.5
Minimum Torque (nN.m) Steady Shear	5
Maximum Torque (mN.m)	200
Torque Resolution (nN.m)	0.05
Minimum Frequency (Hz)	1.E-07
Maximum Frequency (Hz)	100
Minimum Angular Velocity [1] (rad/s)	0
Maximum Angular Velocity (rad/s)	300
Maximum Normal Force (N)	50
Normal Force Sensitivity (N)	0.005
Normal Force Resolution (mN)	0.5

Please define termination steps when you are unsure of the likely range of the results or operating close to the operational limits.

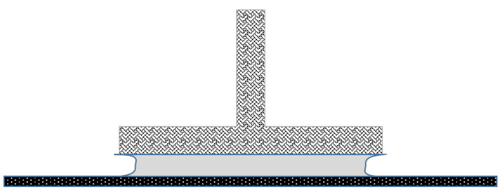


TA instruments also has a library of application notes. Consider reviewing them in preparation for your tests and towards understanding your results. https://www.tainstruments.com/applications-library-search/

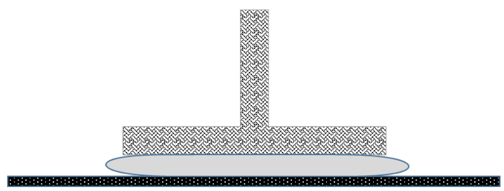
19. Define an end of test step with appropriate ending condition, especially when working with samples that have solidified (increase temperature to melting point)



20. If intending to use the environment chamber, install the border ring on the bottom Peltier plate.
21. Load the sample in the center bottom plate. Consider adding ~10-15% more than the recommended value in the geometry tab (excessive sample can make the cleaning process a hassle) Burst any trapped bubbles in the sample using a needle. Solid samples (like fats, waxes etc.) should be loaded at or beyond their melting point. (turn ON the lamp for better visibility)
22. Click on trim gap (or use the front panel button ). As the geometry makes contact and reaches the trim gap, some of the sample should be squeezed out from under the geometry. Visually verify that the sample is filled uniformly under the geometry. Trim away or wipe down excess sample around the geometry. (Recommendation: use a Kimwipe around a glass slide to wipe low viscosity samples. For viscous samples use the provided tool to move the excess sample away from the geometry and then wipe it). While removing the excess samples, ensure not to scratch the surface or apply much force against the geometry. Drag the sample in simple clean strokes to avoid transferring sample above the test/contact area. Dispose the waste appropriately (biologicals in yellow biohazardous bin, sharps in the yellow sharps container)



After trimming at trim gap



At test gap position

23. Go to test gap position by clicking the geometry gap. If setting up an environment chamber, install the two semi circles metal plates here.

24. Click the start button (software or front panel) to begin the experiment.

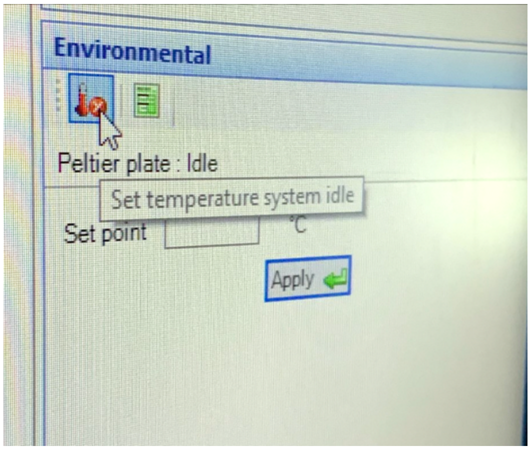
    Note: In some cases where the experiment is not saved properly or set up properly an error message can pop up in the bottom pane on TriOS. While some messages can be traced to a specific error in the experiment set up, most times the error message is relatively vague to decipher. Review the experiment or go back to an experiment that worked for you and build on it. If neither helps, please reach out to the trainer.

Results and unloading

25. Once the system starts recording results, the results screen tab should open with the graphical plot being generated in real-time. Add or change variables to observe on axis of the plots, or change scale by right clicking against the axis definition. Any custom definitions or equations can also be plotted, if defined.
26. In a separate tab, a spreadsheet corresponding to the test results is also being appended as the experiment progresses. Add or change variables displayed in the spreadsheet by choosing select variables on the top left. Any custom definitions or equations can also be recorded, if defined.
27. All experiment graphs tend to be overlaid in one graph, but can be separated out into an independent test graph by right clicking against the experiment in the results tab in the left pane and selecting 'send to new graph'
28. Save or export the results to appropriate file format.

Cleanup

1. If the sample is solidified, set it to the appropriate temperature to melt it.
2. Retract head to a high enough position to retrieve the geometry without interference.
3. Hold the lock button to hear the beep. Hold the geometry, pushing it slightly upwards against the head and slowly unscrew the rod. Remove the geometry and wipe it clean with ethanol and water.
4. Clean the bottom plate with ethanol and water. Ensure no sample debris remains on the instrument or the table.
5. Set the instrument to *Idle* by clicking the thermometer icon in the **Environmental** menu (right side of the software).



6. Click START/STOP to turn off the chiller, it should return to STANDBY mode.

7. Close the software and log out from LMACS.

Troubleshooting

1. If the software closes or crashes: after calibrating the geometry already, there is no need to recalibrate again if the geometry (or accessories) have not been physically changed.
2. If the rheometer screen flashes, there is a communication issue with the instrument and software. Try restarting the software.