

Rheometers – Quick Start Guide
Polymer Facilities - MRL @ UCSB
Rachel Behrens (rachel@mrl.ucsb.edu)

Reservations: Reserve with FBS, login to FBS to access instrument.
Record instrument time on Log sheet.

Safety: The rheometer contains a high-torque motor. Turning on the motor while in dynamic mode causes the motor to snap to dynamic zero position at a high velocity. This can cause severe damage to the transducer and/or personal injury. To avoid damaging yourself and the transducer:

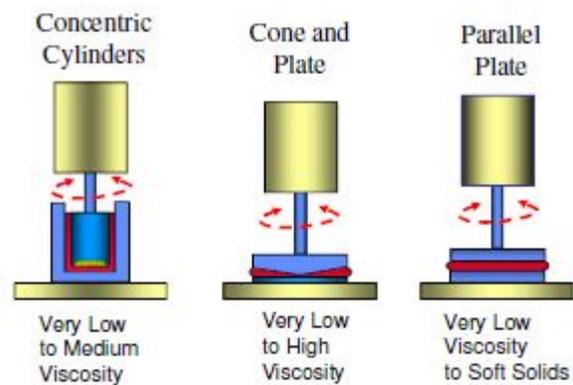
1. Never turn on the motor while a sample is loaded.
2. Keep hands clear of the motor.

Turning on the Instrument:

1. Turn on lab air on the east hood to the red mark on the gauge on Rheometer I (or past if both rheometers are being used)
2. Turn on switch to rheometer on the back of the instrument
3. Turn on water bath and set temperature (for Rheo I)
4. On the computer, open TA Orchestrator

Choosing your parameters:

- Plate geometry and size
 - Plates (8mm, 25mm, 50mm)
 - Cone (25mm, 50mm)
 - Couette
- Test mode and Motor mode: steady or dynamic
 - Dynamic Mode (sinusoidal)
 - Steady Mode (linear)



Choosing a Geometry Size:

1. Assess the 'Viscosity' of your sample.
2. Lower viscosity → larger plate diameter
 - a. Low viscosity (milk)- 50 mm geometry
 - b. Medium viscosity (honey)- 25 mm geometry
 - c. High viscosity (caramel)- 8 mm geometry
3. Examine data in terms of absolute instrument variables [torque/speed/displacement] and modify geometry choice to move into optimum working range.
4. You may need to reconsider your selection after the first run.

Upper Tool Installation:

1. To install an upper tool (i.e. plate, cone), loosen the knobs on the upper arm and insert the tool into the upper portion, with the notches on the tool fitting into the cross bar on the upper arm.
2. Push upper tool fully upward into anvil and hold upper tool in place.
3. Hand tighten the knobs - do not over-torque them.
4. Check that the upper tool is not loose, and there are no gaps.

Zeroing the Gap: Prior to loading your sample, you will need to zero the gap.

1. In TA Orchestrator, open the Instrument Control Panel through Control → Instrument Control Panel
 - a. Change temp. 25°C, temp. control- bath
 - b. Motor- ON
 - c. OK
2. Open the Gap Control Panel screen through Control → Gap Control Panel
 - a. Raise the stage to maximum height by clicking Send to Top. (DO NOT ZERO INDICATOR AT TOP)
 - b. Click Offset Torque to Zero.

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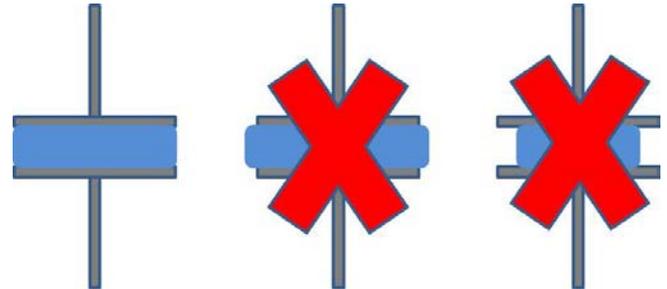
- c. Click Offset Force to Zero.
- d. In the set gap box, set the gap to 5mm.
- e. Once the gap is set, click on Zero Fixture.
- f. After gap is zeroed, set gap to 25mm. Exit to close Gap Control Panel.
- g. Reopen Instrument Control Panel, turn off motor. OK.

Choosing Motor Mode: Choose Motor Mode Prior to Loading Sample

1. Steady: Select Control→Motor Mode: Steady to set the actuator to steady mode.
2. Dynamic: Select Control→Motor Mode: Dynamic to set the actuator to dynamic mode.

Loading sample onto bottom plate (see figure at right)

- With motor off, place enough sample to fill the gap (~ 1 mL depending on gap size)
- Turn on motor (Control→Instrument Control Panel)
- Set gap (Control→Gap Control Panel)
- Trim sample as necessary, so that sample fills the gap



To Edit or Begin the Experiment

- Select **Edit/Start Test** from the **Control** menu or click the  button on the tool bar to begin the process of test execution

Choosing your Test Mode:

Viscosity: Steady Rate Sweep Test on OIL. Plate and bath – 50 mm.

Rate Sweep applies steady shear deformation at user-commanded shear rates. Data can be collected in either time-based mode (one measurement is taken at user command). Rate can be either incremented or decremented. Rate increments can be scaled either logarithmically, discretely, or linearly. (See user manual for definitions.)

1. Enter the desired Gap in mm (2.0), trim sample and change gap = 1.9 mm. Sample should fill the gap.
2. Test Setup- Steady Rate Sweep Test
3. * Strain Controlled *Steady. Edit Geometry- plates, etc., the read test fixture gap- the instrument reads the gap immediately prior to start a test.
4. Edit Test- Temp. 25°C, Sweep Mode *log, Initial Rate $\dot{\gamma}^*$ = 10 1/s, Final Rate $\dot{\gamma}^*$ = 100 1/s, Points per Decade 5, [] Max 500, Mode *time based, delay 5 s, measurement time 10, direction *clockwise, options-delay off.
5. Begin Test
6. Generate an X-Y plot of (choose layout of the plot-left mouse on the plot, change x and y). Y should be $\Delta\eta$ (P) and x: time or rate [s^{-1}]. Where: η = viscosity = $\tau/\dot{\gamma}$, τ = shear stress, $\dot{\gamma}^*$ = strain rate (shear rate).

G G' Crossover: Dynamic Freq. Sweep Test on PDMS. Plate 25 mm + bath 50 mm.

Dynamic Frequency Sweep applies a sinusoidal strain of constant peak amplitude over a range of frequencies. The peak amplitude of strain is determined by the STRAIN command. One measurement is taken at each of the selected frequencies.

1. Instrument Control Panel (change temp. 30°C, temp. control- bath, Motor- ON).
2. Place the sample on to lower fixture (small ball).
3. Enter the desired Gap in mm (2.0), trim sample and change gap = 1.9 mm. Sample should fill a gap.
4. Test Setup- Dynamic Freq. Sweep Test- Edit Test* Strain Controlled *Dynamic. Edit Geometry- plates etc, the read test fixture gap- the instrument reads the gap immediately prior to start a test.
5. Edit Test- Temp. 30°C, Strain 15%, Sweep Mode *log, Initial Freq. ω = 1.0 or 10.0 rad/s, Final Freq. = 100 rad/s. Points per Decade = 5, [] Max 500, Mode *time based, delay 5 s, measurement time 10, direction *clockwise, options-delay off, pre shear-off, auto strn.- off..
6. Begin Test

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7. Generate an X-Y plot (choose layout of the plot-left mouse on the plot, change x and y). Y should be G' and G'' On the left side- and η^* [P] and δ on the right side. X = ω (rad/s).
8. Go to Analysis- G'/G'' Crossover point (stamp).
9. Where- G' = Elastic (storage) Modulus, G'' = Viscous (loss) Modulus, ω = Frequency (angular in rad/sec), $\tan \delta = \text{loss tangent} = G''/G'$.

Cleaning up and Shutting Down the Instrument

1. Turn off motor before removing top tool (Control→Instrument Control Panel)
2. Exit Software
3. Transfer data from hard drive to Z: drive
4. Remove samples from plates using solvents. DO NOT SCRAPE PLATES WITH METAL INSTRUMENTS THAT CAN SCRATCH OR GOUGE THE PLATES
5. Turn off instrument
6. Turn off lab air
7. Log out of instrument in FBS
8. Record actual time on Log Sheet