Manual

Krystyna Brzezinska (kbrzez@mrl.ucsb.edu), 11/01/2005

Please ask for training to get a password.

Rheometer is a mechanical spectrometer that is capable of subjecting a sample to either a dynamic (sinusoidal) or steady (linear) shear strain (deformation), then measuring the resultant torque expended by the sample in response to the shear strain. Shear strain is applied by the motor; torque is measured by the transducer.

Dynamic Mode- the motor begins all tests at the motor zero position, and drives symmetrically about motor zero to the strain commanded by the software. The motor is labeled with graduations indicating 0.1, 0.25, and 0.5 radians from motor zero. The maximum angular deflection of the motor is 0.5 radians from either side of motor zero.

Steady Mode- the motor can begin a test from any position, rotating clockwise or counterclockwise as specified by the software.

Choosing a Geometry Size:

1. Assess the 'Viscosity' of your sample.
2. When a variety of cones and plates are available, select diameter appropriate for viscosity of sample
- low viscosity (milk)- 60 mm geometry
- medium viscosity (honey)- 40 mm geometry
- high viscosity (caramel)- 20 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.

OVERVIEW

Geometry	**Application**	**Advantage**	**Disadvantage**
Cone/plate	Fluids, melts Viscosity > 10 mPas	True viscosities	Temperature ramp difficult
Parallel Plate	Fluids, melts Viscosity > 10 mPas	Easy handling, temperature ramp	Shear gradient across sample
Couette	Low viscosity samples <10 mPas	High shear rate	Large sample volume
Double Wall Couette	Vary low viscosity < 1 mPas	High shear rate	Cleaning difficult
Torsion Rectangular	Solid polymers, composites	Glass to rubbery state	Limited by sample stiffness.

I. Concentric Cylinders (Very low to Medium Viscosity-use for coarse slurries and unstable suspensions)

- Advantages: Large surface area to obtain low stress measurements, good for testing suspensions with limited stability, good for testing samples with large particle size.
- Disadvantages: Possible shear history effects from loading, cleaning can be difficult.

II. Cone and Plate (Very Low to High Viscosity-use for all unfilled liquids, or dispersions (suspensions and emulsions) with small particles or droplets no larger than 1/10th geometry gap)

- Advantages: homogeneous shear, shear rate and stress in the gap, small sample volume (ca. 1 ml), viscosities > 10mPas, cleaning easy, ideal for normal force measurements.
- Disadvantages: highly sensitive to correct positioning (gap) of cone and plate, gap match temperature dependent, sample preparation for high viscosity samples difficult, not suitable for dispersions with solid particles as big as gap, instabilities in shear field can occur at high rates (liquid shear-field breaks up due to centrifugal force).

III. Parallel Plate (Very Low Viscosity to Soft Solids-for filled systems with particles too large for cone and plate).

- Advantages: Variable sample thickness (recommended 0.5 to 2 mm), shear rate limits adjustable by changing gap H or plate diameter R, temperature ramps / sweeps easy to carry out, easy sample preparation for high viscosity samples, small sample volume, viscosities > 10mPas, wall slippage problems can be overcome by using serrated plates.
- Disadvantages: Shear rate not constant (can be corrected), instabilities in shear field can occur at high rates (liquid shear-field breaks up and liquid is lost due to centrifugal force).

IV. Torsion Rectangular (Very Soft to Very Rigid Solids).

- Advantages: High modulus samples, small temperature gradient, simple to prepare.
- Disadvantages: No pure Torsion mode for high strains.

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 or linearly.

a.) Open software- TA Orchestrator
b.) Instrument Control Panel (change temp. 25˚C, temp. control- bath, Motor- ON.
c.) Ensure that a sample is not loaded and the upper and lower fixtures are clean.
d.) Using the Stage Control on the Instrument Panel screen, lower the stage to achieve a Gap of about 0.5 mm (as judged visually).
e.) Select the Gap Control Panel function. The Gap Control dialog box is displayed. Click
- Offset Torque to Zero.
- Offset Force to Zero.
- Gap- Zero Fixture (the stage lowers to achieve contact between fixtures, current gap is zeroed, and the information form is no longer displayed.

f.) Raise the stage to maximum height by clicking Send to Top. Turn off the motor.
g.) NEVER TURN ON MOTOR WHILE A SAMPLE IS LOADED. KEEP HANDS CLEAR OF THE MOTOR. Place the sample on to lower fixture.
h.) Enter the desired Gap in mm (2.0), trim sample and change gap = 1.9 mm. Sample should fill a gap.
i.) Test Setup- **Steady Rate Sweep Test**
j.) * Strain Controlled *Steady. Edit Geometry- plates etc, the read test fixture gap- the instrument reads the gap immediately prior to start a test.
k.) Edit Test- Temp. 25˚C, Sweep Mode *log, Initial Rate γ^* = 10 1/s, Final Rate γ^* = 100 1/s, Points per Decade 5, [] Max 500, Mode *time based, delay 5 s, measurement time 10, direction *clockwise, options-delay off.
l.) Begin Test
m.) 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 = τ/γ, τ= shear stress, γ^* = strain rate (shear rate).

Dynamic Freq. Sweep Test on PDMS (calibration kit). 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.

a.) Open software- TA Orchestrator
b.) Instrument Control Panel (change temp. 30˚C, temp. control- bath, Motor- ON.
c.) Ensure that a sample is not loaded and the upper and lower fixtures are clean.
d.) Using the Stage Control on the Instrument Panel screen, lower the stage to achieve a Gap of about 0.5 mm (as judged visually).
e.) Select the Gap Control Panel function. The Gap Control dialog box is displayed. Click
- Offset Torque to Zero, Offset Force to Zero.
- Gap- Zero Fixture (the stage lowers to achieve contact between fixtures, current gap is zeroed, and the information form is no longer displayed.

f.) Raise the stage to maximum height by clicking Send to Top. Turn off motor. NEVER TURN ON MOTOR WHILE A SAMPLE IS LOADED. KEEP HANDS CLEAR OF THE MOTOR Place the sample on to lower fixture (small ball).
g.) Enter the desired Gap in mm (2.0), trim sample and change gap = 1.9 mm. Sample should fill a gap.
h.) Test Setup- **Dynamic Freq. Sweep Test**- Edit Test
i.) * Strain Controlled *Dynamic. Edit Geometry- plates etc, the read test fixture gap- the instrument reads the gap immediately prior to start a test.
j.) Edit Test- Temp. 30˚C, Strain 15%, Sweep Mode *log, Initial Freq. $\omega = 1.0$ 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..
k.) Begin Test
l.) 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).
m.) Go to Analysis- G'/G" Crossover point (stamp).

Where- G'= Elastic (storage) Modulus, G"= Viscous (loss) Modulus, ω= Frequency (angular in rad/sec), tan δ = loss tangent = G"/G'.