BROOKFIELD

Operation Manual
R/S SST2000
Soft Solid Tester

1788Lab INSTRUMENT

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Operation Manual

knowledge

Action movie

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Operating The SST2000 Soft Solids Tester

Running a sample using software control

1. In Measure/Analysis click Load Program File and select the required test method.
2. Centre the sample on the base plate and fix in place with the clamps.
3. Lower the instrument head by pushing down on the "periscope handles" so that the vane enters the sample to the required depth.
4. Click Start, enter the required filename for the results and click OK.
5. Enter any other details relating to batch numbers, customer, operator etc. and click OK.

The measurement will start.

Standard graphing functions and results overlay procedures are covered in the Rheo2000 software manual and the onboard Help file.

Soft Solids Analysis Functions

Clicking on Soft Solids Analysis opens the analysis functions for soft solids testing.

The Soft Solids Analysis Functions enable the quantification of properties of tested materials using regression analyses and peak-value identifications:

<u>Yield / Modulus</u>

The peak value within the data range is reported as *Yield Point* values for *Stress* and *Torque*. The *Strain* and Angular *Displacement* at the yield point are also reported.

A regression is performed on the range defined in <u>Configuration</u> and the gradient of the stress vs. strain curve is reported as *Modulus*. The regression coefficient, B, and the standard deviation, S, is also reported.

<u>Creep</u>

Two regressions are performed on the creep data to obtain:

Instantaneous Response: A regression is performed on the range defined in <u>Configuration</u> to obtain values for instantaneous *Compliance*, *Strain* and angular *Displacement*.

Viscous Response: A regression is performed on the range defined in <u>Configuration</u> to obtain *Creep Viscosity.* The intercept on the y axis (strain, Displacement or Compliance) is then divided by the maximum value (last data point) of the creep curve to obtain *Elastic Index*

Configuration

Modulus: The upper and lower limits of the range for the modulus regression is defined here as percentages of the peak value. A typical range is 20% to 70% but these should be altered to accommodate longer or shorter linear regions.

Creep: The number of points for the regressions for Instantaneous Response and Viscous Response are entered here. Typical values are 5 points and 20 points respectively.

Running a sample in Stand-Alone mode.

Running a one-off test

1. When flashing cursor is on Run Single press OK.
2. Select the measuring system in use
3. Scroll the cursor down to the selected test method: Yield Test or Creep Test.

For Yield Test:

4. Set Rate (rotational rate in rpm), Nr. Of MP (the number of measuring points to be taken) and Time (the total time of the test).
5. Input any required identification for the data file eg batch number, operator etc. and press OK
6. Press ST to Start the test.

The display will then show the current and peak values for stress (in Pascals) and torque (in milliNewtonmetres):

```
Stress (Pa): 00000
Peak: 00000
Torque (mNm): 00.00
Peak: 00.00
```

For a Creep Test:

4. Set Stress (the applied stress in Pascals), Nr. Of MP (the number of measuring points to be taken) and Time (the total time of the test).
5. Input any required identification for the data file eg batch number, operator etc. and press OK
6. Press ST to Start the test

The display will then show the Angular Position and Instantaneous Angle (in milliradians), Time (in seconds) and Creep Rate (in milliradians per second):

```
Angle (mrad): 0000
Inst. Angle: 0000
Time (s): 0000
Rate (mrad/s): 000
```

Editing and Running Programs
As detailed in the RS Rheometer operation manual, stand-alone programs can be written under the Utilities → Edit Programs menu for the SST2000 to facilitate fast test turnaround.

Getting Started In Soft Solids Testing

Two test methods are most often used to evaluate soft solids with the SST2000: The Constant Rate Test and The Creep Test.

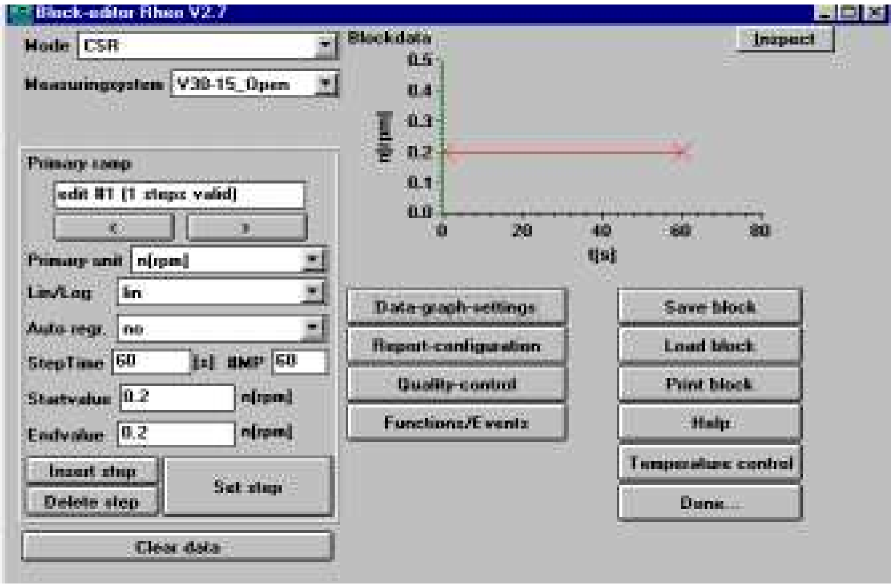
The Constant Rate Test

For products like: Stiff pastes, slurries, set gels, waxes.

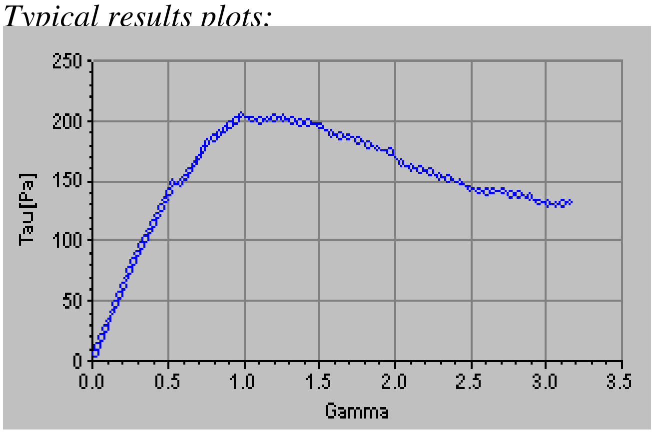
Properties measured: Yield stress or torque, equilibrium stress strain at yield, rigidity modulus.

Description: In the constant rate test the vane is rotated at a constant low rotational rate (typically 0.1 to 0.5 rpm) and torque or stress is measured against time, rotational angle or strain.

Example method: Constant rotation at 0.2 rpm, linear data collection, 60 data points over 60 seconds. A V30-15 vane (30mm high by 15mm across) is used in an open configuration (see Setting Up Vanes).



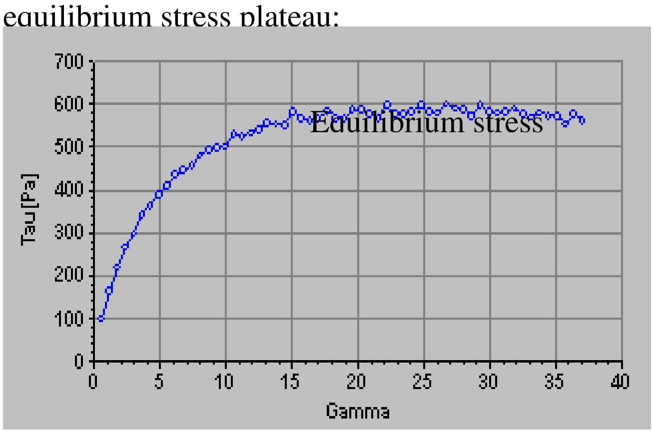
Typical results plots:



CR Test on mayonnaise with stress (Tau, in Pascals) plotted against strain (Gamma, unitless).

The peak value gives us yield stress. The gradient of stress/strain upcurve indicates gel rigidity, a steeper curve: a stiffer gel and vice-versa.

Mayonnaise shows a distinct gel breakdown after the peak yield point. Other samples, such as some pastes and slurries may not peak but equilibrate at an equilibrium stress plateau:



CR test on toothpaste. Equilibrium stress is an indication of low shear viscosity or "consistency".

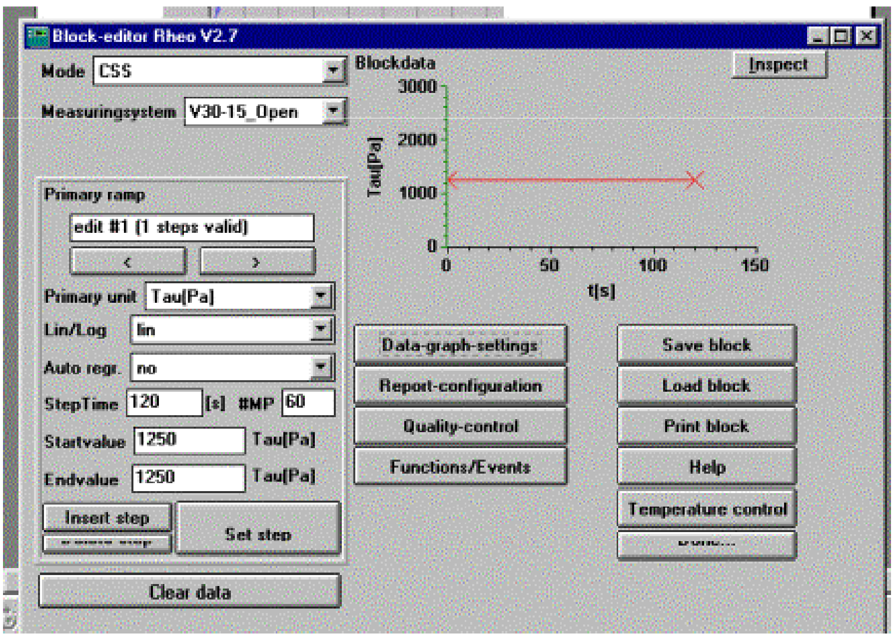
The Creep Test

For products like: More mobile gels and lumpy products : custard, gravies and sauces, jams and marmalades.

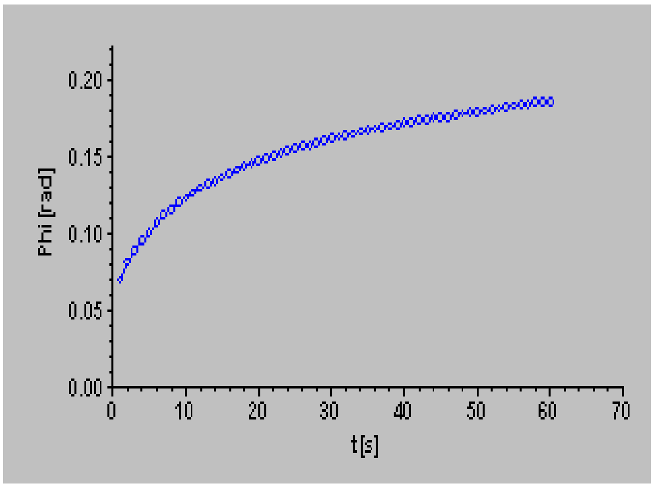
Properties measured: Instantaneous compliance/strain, low shear viscosity/creep rate, elasticity index.

Description: A single stress/torque is applied at time t=0 and maintained on the sample over a period of time, usually between one and five minutes. The degree of subsequent movement in the sample, is recorded against time in the form of angular displacement (in radians), strain (unitless) or compliance (strain divided by applied stress – in m^2/N).

Example method: A constant stress of 1250Pa is applied over a period of 120 seconds. 60 data points are collected over this period.



Typical results plot:



Creep test on fruit conserve. Angular displacement (Phi, radians) is plotted against time. Short timescale response relates to elastic behaviour and long timescale response relates to viscous behaviour. So a high initial angular displacement indicates a flexible "wobbly" gel structure while a steep gradient at long timescales means a product that creeps easily, that is, it has a low low-shear viscosity.

A Few Tips To Get You Started

It is important to operate the SST2000 at torque values that are within the SST2000's reliable range of 30‰ (3% or 1.5mNm) to 1000‰ (100% or 50mNm). In creep testing the minimum recommended applied torque is 75‰ (3.75mNm).

If you prefer to work in stress values then these torques equate to the following for our standard vanes:

	Constant Rate Test	Creep Test (recommended minimum)
V30-15	121 Pa to 4040 Pa	303 Pa
V40-20	51 Pa to 1700 Pa	128 Pa
V80-40	6.4 Pa to 213 Pa	16 Pa

Vane selection

The first consideration in vane selection is the working torque ranges just mentioned. The next consideration relates to vane-to-container diameter ratios. The vane measuring method works best when these ratios are lower than 0.75. Low vane/container ratios allows us to measure samples with large particulates dispersed throughout and we can also apply low strains to our sample while still operating at angular displacements well within the instrument range.

Ideally, the vane should be immersed in a sample of a depth twice that of the vane height. This is, however, very often not practical but the user should at least ensure ample clearance between the lower end of the vane and the base of the container, especially when measuring large-particle suspensions.

Container Dimensions

For yield point measurements the container dimensions are largely irrelevant. For creep tests and strain or modulus measurements in constant rate tests the container diameter does come into effect, especially with higher diameter ratios, and must therefore be entered into the measuring system setup details in the Meas-Editor utility program. For diameter ratios of less than 0.2 minor changes in the container size, design or positioning can be more or less ignored for most applications.

Setting Up Vanes In Measuring System Editor

Before you can use a vane it's details must be set up in Meas-Editor.

1. Open Meas-Editor
2. Click New
3. Enter the name of the vane (eg V80-40) and select Type → Vane.
4. Enter the factors K-Gamma and Tau_Prom that were supplied with the vane.
5. Enter the radius of the vane (Radius i) and the radius of the container (Radius o).
6. Click Save.
7. Repeat as necessary and close Meas-Editor.

Vane Constants For The Soft Solids Tester

Stress Constant: $\tau_{Prom.}$

All standard vanes supplied for the Soft Solids Tester have a height (H) to diameter (D) ratio of 2:1. A stress constant is required for each vane to convert torque in Newton metres to shear stress in Pascals. This constant is calculated as follows:

$$\tau_{prom} = \frac{10^{-4}}{\pi D^3}\left(\frac{H}{D} + \frac{1}{3}\right)^{-1}$$

The constants for our standard vanes are as follows:

Vane	Diameter (m)	Height (m)	τ-prom
V30-15	0.015	0.030	4.043
V40-20	0.020	0.040	1.706
V80-40	0.040	0.080	0.2132

These are now pre-loaded in our software. If you do not have these vanes available in Block Editor or on your instrument then please load them using Meas-Editor for software driven setups, or Utilities → Measuring Systems for stand-alone setups.

Strain / Rate Constant: K-Gamma

The constant K-Gamma converts the rotational rate or position into shear rate / strain values. It's value is dependent upon the ratio of container-to-vane diameter. The vanes pre-loaded into your software (eg V40-20-3to1) assume a ratio of 3:1. If you use vane/container combinations that don't match this you will need to set up a new measuring system in Meas-Editor or Utilities → Measuring Systems. You can calculate out the required value using:

$$K - gamma = \frac{0.2094}{1 - b^2}$$

where b is the ratio of *inner to outer* diameters, or select an appropriate value from the following table:

Container to Vane Diameter Ratio	K-Gamma
1.5 to 1	0.3763
2 to 1	0.2792
3 to 1	0.2355
4 to 1	0.2234
5 to 1	0.2181
Infinite sea	0.2094