BROOKFIELD DV-III ULTRA

PROGRAMMABLE RHEOMETER

Operating Instructions

Manual No. **M/98-211-B0104**

Please record the Model and Serial Number of your viscometer. Having this information readily available will help us to assist you should there be any questions regarding your instrument.

Model No. ________________________

Serial No. ________________________

Use of this instrument in a manner not specified by Brookfield may result in incorrect readings or instrument failure. Please read this manual prior to using the instrument.

BROOKFIELD

SPECIALISTS IN THE MEASUREMENT AND CONTROL OF VISCOSITY

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Operation Manual

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Action movie

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I. INTRODUCTION

The Brookfield DV-III Ultra Programmable Rheometer measures fluid parameters of Shear Stress and Viscosity at given Shear Rates. Viscosity is a measure of a fluid's resistance to flow. You will find a detailed description of the mathematics of viscosity in the Brookfield publication "*More Solutions to Sticky Problems*", a copy of which was included with your DV-III Ultra.

The principle of operation of the DV-III Ultra is to drive a spindle (which is immersed in the test fluid) through a calibrated spring. The viscous drag of the fluid against the spindle is measured by the spring deflection. Spring deflection is measured with a rotary transducer. The viscosity measurement range of the DV-III Ultra (in centipoise or cP) is determined by the rotational speed of the spindle, the size and shape of the spindle, the container the spindle is rotating in, and the full scale torque of the calibrated spring.

The DV-III Ultra can also measure yield stress (in Pascals or Pa). See Section I.3 for more information on yield stress.

I.1 Torque Measurement

There are four basic spring torque modelsoffered by Brookfield:

	Spring Torque	
Model	dyne•cm	mN•m
LVDV-III Ultra	673.7	0.0673
RVDV-III Ultra	7,187.0	0.7187
HADV-III Ultra	14,374.0	1.4374
HBDV-III Ultra	57,496.0	5.7496

The higher the torque calibration, the higher the viscosity measurement range. The viscosity measurement range for each spring torque model may be found in Appendix B.

I.2 Viscosity Units of Measurement

All units of measurement are displayed according to either the CGS system or the SI system.

1. Viscosity appears in units of centipoise (shown as "cP") or milliPascal-seconds (shown as mPa•s).
2. Shear Stress appears in units of dynes/square centimeter ("D/cm^2") or Newtons/square meter ("N/m^2").
3. Shear Rate appears in units of reciprocal seconds ("1/SEC").
4. Torque appears in units of dyne-centimeters or Newton-meters (shown as percent "%" in both cases).

The equivalent units of measurement in the SI system are calculated using the following conversions:

	SI		CGS
Viscosity:	1 mPa•s	=	1 cP
Shear Stress:	1 Newton/m^2	=	10 dyne/cm^2
Torque:	1 N•m	=	10^7 dyne•cm

References to viscosity throughout this manual are done in CGS units.

I.3 Yield Stress Measurement

Another feature now available in the DV-III Ultra Rheometer is the yield stress test.

The yield point is the point at which a material begins to flow. The associated properties are the yield stress and yield strain. The yield stress is the critical shear stress, applied to the sample, at which the material begins to flow as a liquid. The yield strain is the deformation in the material, resulting from the applied stress prior to the start of flow.

Many materials are designed to have a yield point, so that the behavior of their products satisfies various customer needs. Foods often have yield points. Ketchup in particular must flow out of a bottle when shaken or squeezed, but then solidify on the targeted food such as french fries. Shaking or squeezing the bottle stresses the ketchup so that it flows; after the ketchup settles on the fries, its structure rebuilds so the ketchup "sits" in place rather than flowing off the fries like water. Puddings have yield points, as well. The "body" of the pudding appeals to consumers – it is solid at rest, yet it's easily spooned out of its cup and is easy to eat. Thus, the yield behavior of many foods contributes to the food texture that we like.

Many paints have low yield stresses. Many latex house paints, for example, are easily stirred or poured. Brushing or spraying provides enough stress so that the paint flows easily and smoothly over a painted wall. However, a thin layer of applied paint (if a good one!), allowed to rest undisturbed on the surface, regains its structure quickly so that there is very little unsightly "dripping" afterwards. The smooth appearance of the painted surface is very appealing to the homeowner.

The operating principle is to drive a vane spindle through the calibrated spiral spring connected to a motor drive shaft (see Figure I-1). The vane spindle is immersed in the test material. The resistance of the material to movement is measured by observing increasing torque values as the DV-III Ultra motor rotates. The amount of shaft rotation is measured by the deflection of the calibrated spiral spring inside the instrument. Spring deflection is measured with a rotary transducer.

If the vane spindle did not move at all, the data would look like the graph in Figure I-2. The data often looks like the graph in Figure I-3 because there is usually some deformation of the test material due to the increasing force imparted by the vane spindle. The maximum torque value is the yield point. The straight line in Figure I-3 is a repeat of what was shown in Figure I-2. An algorithm in the firmware converts the maximum torque value into a yield stress value.

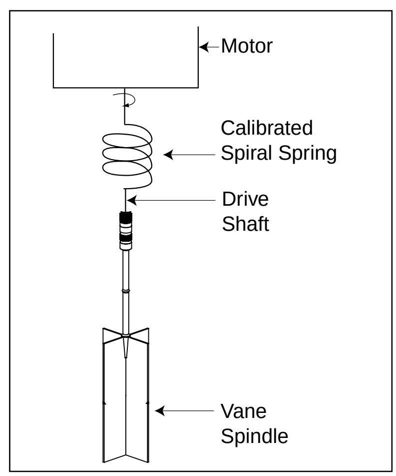


Figure I-1

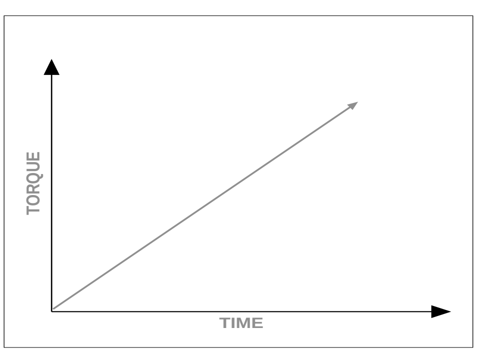


Figure I-2

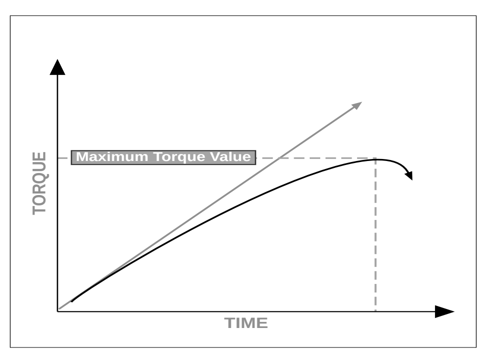


Figure I-3

The shear stress measurement range of the DV-III Ultra (in Pascals) is determined by the size and shape of the vane spindle and the full scale torque range of the calibrated spring.

I.4 Components

Component	Part Number
DV-III Ultra Rheometer	depends on model
Powerbase	DVP-2Y
includes:	
Leveling Screws (3)	VS-3
Upright Rod	VS-20
Jam Nut	VS-21
Clamp Assembly	VS-27Y
Spindle Set with Case	
LVDV-III Ultra set of four spindles or	SSL
RVDV-III Ultra set of six spindles or	SSR
HA/HBDV-III Ultra set of six spindles	SSH
Optional Vane Set of three spindles	SSVANE
For Cone/Plate versions: a spindle wrench, one cone spindle and sample cup Part No. CPE-44Y replace the spindle set.	
Power Cord	
for 115 VAC	DVP-65
for 230 VAC	DVP-66
RTD Temperature Probe	DVP-94Y
Ribbon Cable	DVP-145
Guard Leg:	
LVDV-III Ultra	B-20Y
RVDV-III Ultra	B-21Y
Carrying Case	DVP-71Y
RHEOLOADER Software, EZ-Yield Software	CD-PROGA
Cable (DV-III Ultra to Computer)	DVP-80
Operator Manual	M/98-211

Please check to be sure that you have received all components, and that there is no damage. If you are missing any parts, please notify Brookfield Engineering or your local Brookfield agent immediately. Any shipping damage must be reported to the carrier.

I.5 Utilities

Autosensing Power Supply:

Input Voltage:	90 - 260 VAC
Input Frequency:	50 - 60 Hz
Power Consumption:	Less than 220 UA

Power Cord Color Code:

	United States	Outside United States
Hot (live)	Black	Brown
Neutral	White	Blue
Ground (earth)	Green	Green/Yellow

I.6 Specifications

Speed Ranges for viscosity tests:	0.01-250 RPM, 0.01 RPM increments from 0.01 to 0.99 RPM, 0.1 RPM increments from 1.0 to 250 RPM	
Speed Ranges for yield tests:	Pre Shear	0.01 to 200 rpm
	Zero	0.01 to 0.5 rpm
	Yield Test	0.01 to 5 rpm

Time Intervals for yield tests: 100 msec - 1000 msec

Viscosity Accuracy: ± 1.0% of full scale range for a specific spindle running at a specific speed.

Temperature sensing range: - 100°C to 300°C (-148°F to 572°F)

Temperature accuracy: ± 1.0°C from -100°C to 150°C
± 2.0°C from +150°C to 300°C

Analog Torque Output: 0 - 1 Volt DC (0 - 100% torque)

Analog Temperature Output: 0 - 4 Volts DC (10mv / °C)

Printer Output: Centronics, parallel or serial

Computer Interface: RS232

Torque Accuracy: ±1.0% of full scale range

Torque Repeatability: ±0.2%

Operating Environment: 0 °C to 40 °C temperature range (32 °F to 104 °F)
20% - 80% R.H.: non-condensing atmosphere

Weight:	Gross Weight:	35 lbs.	15.9 kg
	Net Weight:	32 lbs.	14.5 kg
Carton Volume:	2.0 cu. ft.	0.057 m^3	

I.7 Safety Symbols and Precautions

Safety Symbols

The following explains safety symbols which may be found in this operating manual.

Indicates hazardous voltages may be present.

Refer to the manual for specific warning or caution information to avoid personal injury or damage to the instrument.

Precautions

If this instrument is used in a manner not specified by the manufacturer, the protection provided by the instrument may be impaired.

This instrument is not intended for use in a potentially hazardous environment.

In case of emergency, turn off the instrument and then disconnect the electrical cord from the wall outlet.

The user should ensure that the substances placed under test do not release poisonous, toxic or flammable gases at the temperatures which they are subjected to during the testing.

I.8 Data Retention

The DV-III Ultra will save spindle parameters (used to calculate centipoise, shear rate and shear stress), default settings and the test data from the last program test run when the rheometer is turned off or there is a power failure.

I.9 Set-Up

1) Place the upright rod into the hole at the front of the base. The rack gear and clamp assembly should face the rear of the base (see Figure 1). The upright rod is held in place with the jam nut which is attached from the bottom of the base. Tighten this nut with a suitable wrench (spanner).

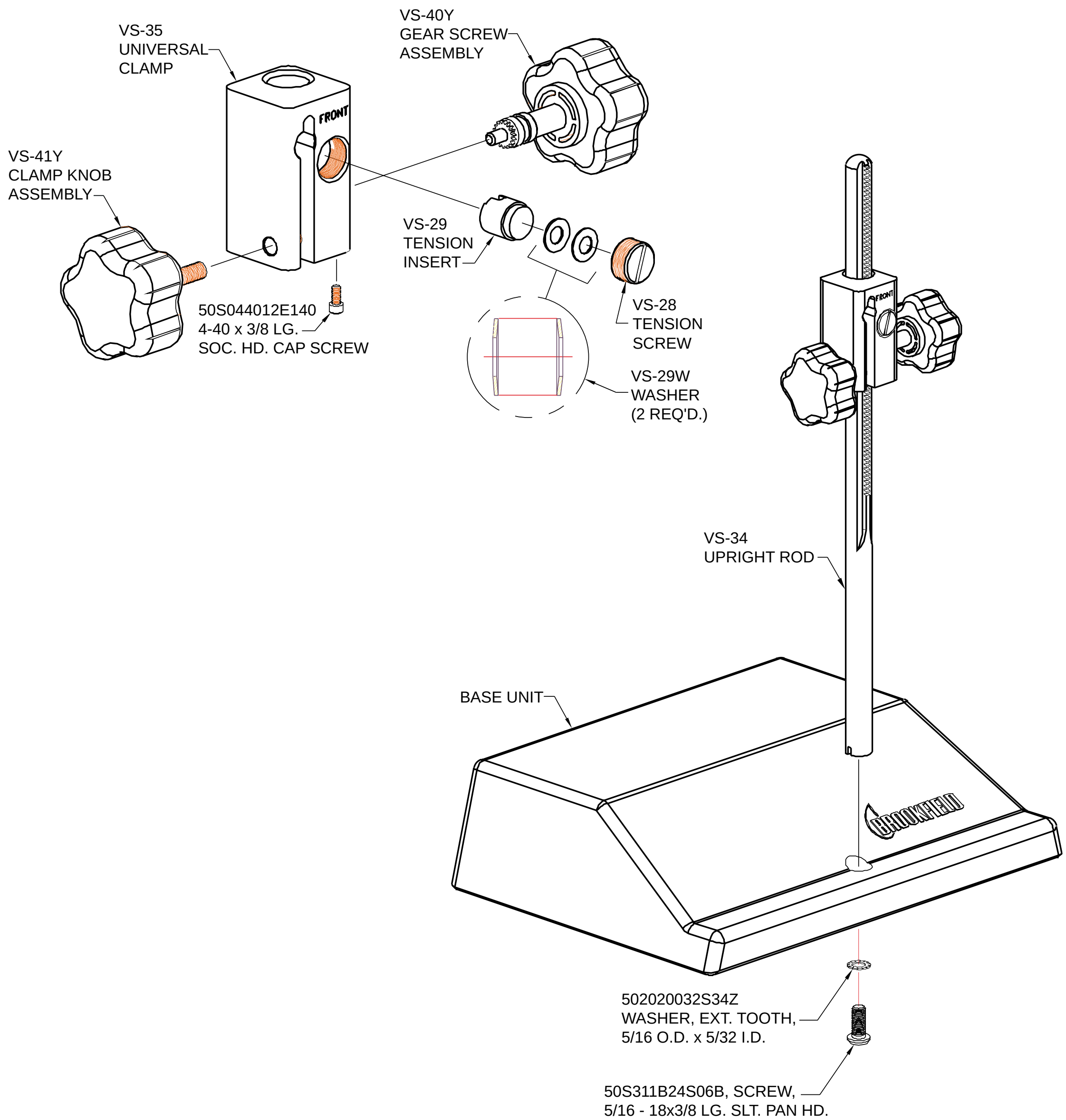


Figure 1

2) Insert the mounting handle on the back of the DV-III Ultra into the hole on the clamp assembly (Figure 2).

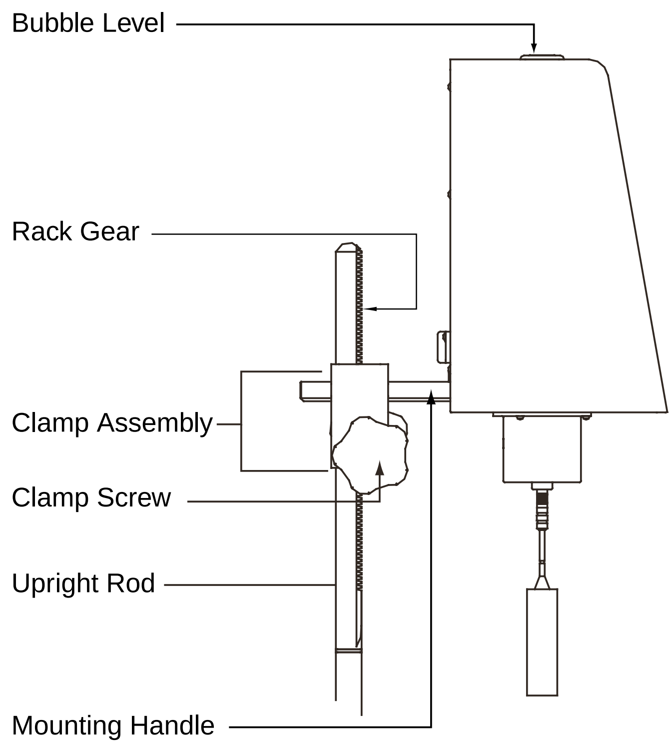


Figure 2

3) Tighten the DV-III Ultra clamp Screw (Figure 2).

> **Note:** If the clamp assembly moves along the upright rod too freely, tighten the tension screw (see Appendix F).

4) Insert the ribbon cable into the DV-III Ultra Rheometer head. Insert the other end of the ribbon cable into the connector on the DV-III Ultra base (see Figure 3).

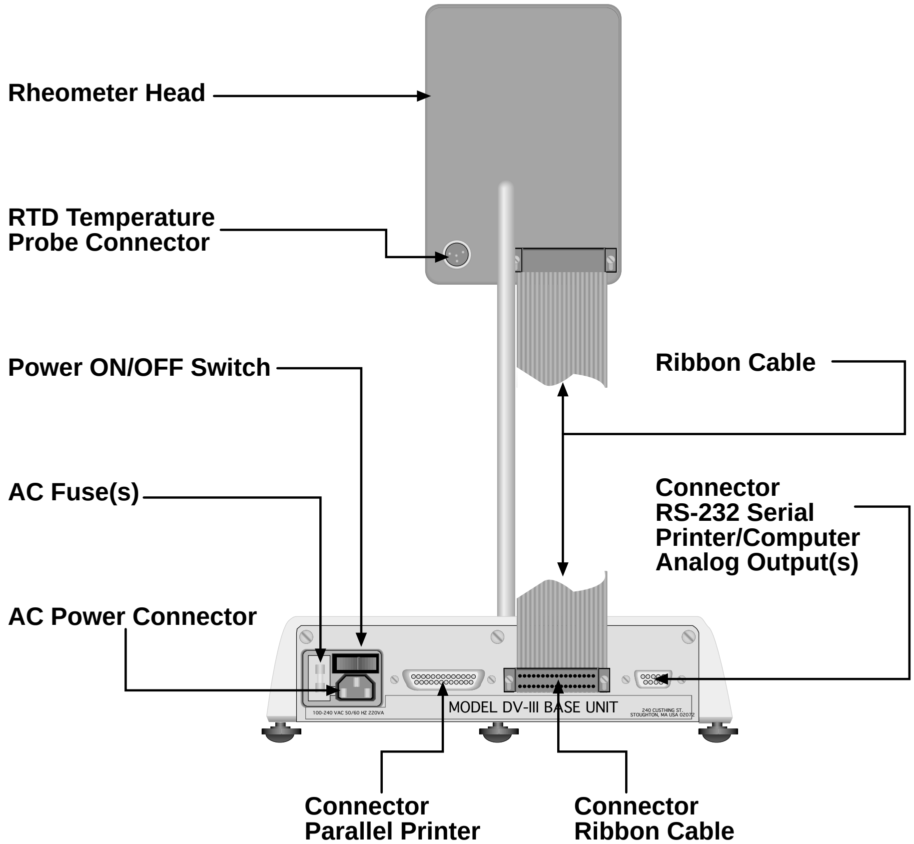


Figure 3

5) Connect the RTD probe to the socket on the back side of the DV-III Ultra Rheometer (Figure 3).

6) The Rheometer must be leveled before the instrument is zeroed and readings are taken. The level is adjusted using the three leveling screws on the base. Adjust so that the bubble level on top of the DV-III Ultra (Figure 2) is centered within the circle.

7) Make sure that the AC power switch at the rear of the base unit is in the OFF position. Connect the AC plug to the socket on the back of the DV-III Ultra base and plug it into the appropriate AC line. Allow at least 10 minutes for warm up; 30 minutes is preferable.

The DV-III Ultra must be earth grounded to ensure against electronic failure!!

8) Temperature monitoring is assured (after the instrument has stabilized) to within ±1.0°C in the range -100°C to +150°C and within 2°C in the range 150°C to 300°C.

9) For Cone/Plate models refer to Appendix A.

10) For printers, software and temperature controllers, refer to Section I.10, Connections.

I.10 Connections

The DV-III Ultra Rheometer is capable of communicating with several external devices to enhance operation. The cables and connections required for proper communication are detailed below.

♦ RHEOLOADER AND EZ-YIELD SOFTWARE

DVP-80 cable is used to connect the RS232 serial port on the DV-III Ultra base to Com Port 1 or Com Port 2 on the computer. Some PCs may also have Com Port 3 and Com Port 4. This cable is supplied with the DV-III Ultra.

♦ RHEOCALC SOFTWARE

DVP-80 cable is used to connect the RS232 serial port on the DV-III Ultra base to Com Port 1 or Com Port 2 on the computer. This cable is supplied with the RHEOCALC software.

♦ PARALLEL PRINTER

CAP-86 cable is used to connect the 25-pin parallel port on the DV-III Ultra base with the Centronics port on the printer.

♦ SERIAL PRINTER

DVP-81 cable is used to connect the 9-pin serial port on the DV-III Ultra with the 25-pin serial port on a printer.

♦ THERMOSEL CONTROLLER, MODEL HT-106
TC-200/TC-500/TC-201P/TC-501P BATH, MODEL HT-107

DVP-141 cable is used to connect the serial port on the DV-III Ultra base to the serial port on the controller. This cable is supplied with the controller/bath.

Be sure that the controller temperature probe is properly located in the control device (Thermosel or bath) and connected to the controller.

Notes:

1. The controller may alternately communicate with Rheocalc V 2.0 or higher software. In this configuration, the controller is connected to the computer through either Com Port 1 or Com Port 2. The DV-III Ultra is also connected to a computer Com Port.
2. The controller must also be connected to the control device (Thermosel or bath) with the appropriate load cable.

♦ STRIP CHART RECORDER

DVP-96Y cable is used to connect the serial port on the DV-III Ultra to the input block of the strip chart recorder. This cable is supplied with a Brookfield strip chart recorder.

I.11 Key Functions

Figure 4 shows the control keys on the face of the DV-III Ultra Rheometer. The following describes each key's function.

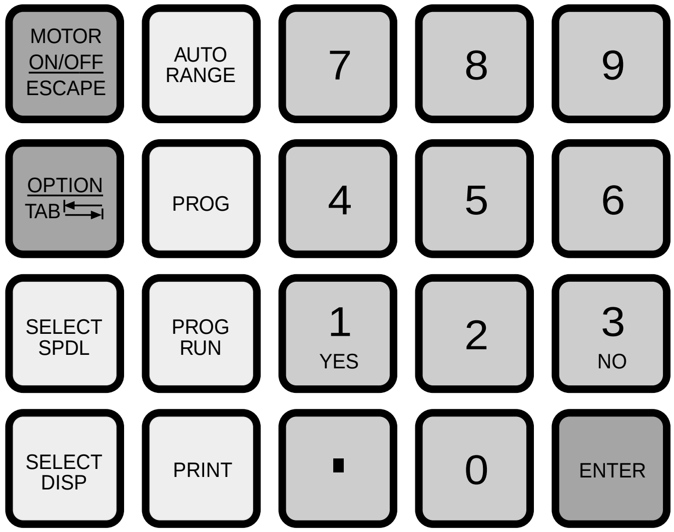


Figure 4

MOTOR ON/OFF, ESCAPE

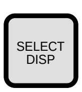
Turns the motor on or off. Cancels any operation, returns the user to the previous screen.

AUTORANGE

Presents the maximum (100% torque) viscosity attainable using the selected spindle at the current speed.

SELECT SPDL

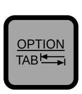
Allows selection of the spindle to be used.



SELECT DISP

Selects the parameter to be displayed:

% Rheometer Torque (%)
cP Viscosity (cP or mPa·s)
SS Shear Stress (Dynes/cm^2 or Newtons/m^2)
SR Shear Rate (1/Sec)



OPTION, TAB

Accesses options menu. See Section V. Toggles between selectable items when indicated.

PRINT

Sends a single line of data to an attached printer. Selects printing and non-printing mode as selected in the Options menu.

PROG

Access the Programs menu for program creation, running or deleting. Contstructs a test program. Allows you to review/modify an existing test program. Execute a Bevis program.

PROG RUN

Execute DV-III Ultra speed/time pair program.

NUMBER KEYS (0 through 9)

Sets speeds and choose items from various dialog screens and the option menu.

ENTER

Functions as an ENTER key similar to a computer by serving to accept a keyboard entry.

I.12 Cleaning

Be sure to remove the spindle from the instrument prior to cleaning. Severe instrument damage may result if the spindle is cleaned in place.

Instrument and Keypad:	Clean with a dry, non-abrasive cloth. Do not use solvents or cleaners.
Immersed Components (spindles):	Spindles are made of stainless steel. Clean with a non-abrasive cloth and solvent appropriate for sample material.

Note: When cleaning, take care not to apply excessive force - it may bend the spindles.

II. GETTING STARTED

II.1 Autozero

Before readings may be taken, the Rheometer must be autozeroed. This is done each time the power switch is turned on. The Rheometer will guide you through the procedure, as follows:

Turn power switch on; as shown in Figure 5, the screen indicates that the DV-III Ultra is in the standalone mode (is not connected to a computer) and gives the version of the operating firmware (the built in program which controls the instrument) and a two-digit alphanumeric code which indicates the Model number (see Table D2 in Appendix D; the code tells the spring torque rating of your Rheometer).

```
     BROOKFIELD
DV-III Ultra RHEOMETER
      V1.0  HA
     STANDALONE
```

Figure 5

No key press is necessary. After a short pause the display will read ***"REMOVE SPINDLE, LEVEL RHEOMETER AND PRESS THE MOTOR ON/OFF KEY TO AUTOZERO."*** Before beginning the autozero procedure, Brookfield recommends that you allow 10 minutes for the instrument to warm up.

After pressing the **MOTOR ON/OFF** key, the screen "flashes" for approximately 15 seconds while the DV-III Ultra autozeros.

After 15 seconds the display reads ***"AUTOZERO IS COMPLETE REPLACE SPINDLE AND PRESS ANY KEY."*** Press a key.

The main screen is displayed and the DV-III Ultra is ready for use (Figure 6).

```
RPM:0.0  SPINDLE:31
TEMP:72.1 F    PRTN

TORQUE = 0.0 %
```

Figure 6

II.2 Rheometer Display

The DV-III Ultra Rheometer is supplied with a 4-line display. The basic set of information is called "The Default Screen" and is shown in Figure 7. The parameters are detailed below:

```
RPM:0.0   SPINDLE:31
TEMP:72.1°F     PRTN

TORQUE = 0.0 %
```

Figure 7

1. **Motor Status and Current Rheometer Speed**
   The DV-III Ultra motor can be OFF, ON at 0.0 rpm or ON at a speed greater than 0.0 rpm. When the motor is OFF, "OFF" will be displayed and no speed entry will be accepted. When the motor is ON, the actual speed of rotation will be displayed. When the motor is switched from ON to OFF, the speed of rotation will be remembered; when the motor is turned ON again, the DV-III Ultra will operate at that same speed. The rheometer motor is set to "OFF" after AUTOZERO.

Note: Motor OFF and a speed setting of 0.0 are essentially the same.

2. **Spindle Number**
   The currently-selected spindle. Viscosity, shear rate, and shear stress values will be calculated based on this number. See Section II.3.

3. **Measured Temperature**
   The current temperature as measured by the attached temperature probe. If no probe is connected, four dashes "----" will be displayed.

4. **Printing Status**
   Indicates the currently-selected method of printing.

5. **Measured Data**
   Instrument Torque (%), Viscosity (cP), Shear Stress (D/cm2), Shear Rate (s^{-1})
   The parameters are toggled from one to another using the Select Display key.

Note: Shear Stress and Shear Rate data cannot be calculated for some spindle geometries. In these cases, the display will show 0.0.

6. **Blank Line**
   This line is used to display entry data when selecting a spindle or speed of rotation. Additionally, selected programs available for running will be identified here when in the Program mode. (See Section IV.2).

The default screen will appear at the completion of the AUTOZERO sequence each time the DV-III Ultra is turned ON in the standalone mode (see Section II.5 for external control mode). The displayed data may be changed as described in the following sections.

The format for data displayed in the default screen and all other screens is described in Table 1. For appearance sake, the entries in the table have been decimal point aligned. Actual rheometer display will have all fields left justified.

Item	Print	Format	Range	Example
RPM	RPM	X.XX XX.X XXX.X	0.01 <= RPM <= 0.99 0.1 <= RPM <= 99.9 100 <= RPM <= 250	0.09 2.4 150.0
Model	M	XXXXX	See Model Table D2 - App D	RV
Spindle	S	XX	00 <= S <= 99	31
Torque	T	XX.X	-10.1 <= T <= 99.9	82.4
Viscosity	cP or mPas	X.XX XXX.X XXXXX XXXeX	0 <= cP <= 9.99 10 <= cP <= 999.9 1000 <= cP <= 99999 100000 <= cP <= 51200000000	3.16 123.8 12345 123e3 to 8
Shear Stress	D/CM2 or N/M2 = D/CM2/10	X.XX XXX.X XXXXX XXXeX	0 <= D/CM2 <= 9.99 10 <= D/CM2 <= 999.9 1000 <= D/CM2 <= 99999 100000 <= D/CM2 <= 999999	4.56 234.5 12345 123e3
Shear Rate	1/SEC	X.XXX XX.X XXXXX	0 <= 1/SEC <= 9.999 10 <= 1/SEC <= 99.9 100 <= 1/SEC <= 99999	1.234 20.7 200
Temperature	T		-99.9 <= T <= 300.0	-10.3
Time	Z	XX:XX	00:00 <= Z <= 99.59	05:32

Table 1

OUT OF RANGE INDICATORS

The DV-III Ultra is capable of measuring instrument torque within the range of 0 to 100%. Based on this measurment, viscosity and shear stress are calculated. Brookfield recommends that data be collected only in the range of 10 to 100%. Any data collected outside of this range is considered invalid.

The DV-III Ultra provides the following display indicators when the measurement point is outside of the 10-100% acceptable range.

TORQUE GREATER THAN 100%

When Rheometer torque exceeds 100%, the parameter display field will show “**EEEE**” for torque, viscosity and shear stress.

```
RPM:112  SPINDLE:31
TEMP:72.1°F    PRTN

TORQUE = EEEE %
```

Figure 8

TORQUE LESS THAN 10%

When Rheometer torque drops below ten (10) percent, the Rheometer will *continue* to display measurement (%, cP, D/cm^2) values with units flashing:

```
RPM:112  SPINDLE:31
TEMP:72.1°F    PRTN

VISC = 900@6   CPS
```

Figure 9

- **TORQUE LESS THAN 0%**

When Rheometer torque drops below zero (0) percent, the Rheometer will *continue* to display torque values preceded by a minus (-) sign. The viscosity and shear stress field will display dashes (- - - -) as indicated in the next screen display:

```
RPM:112  SPINDLE:31
TEMP:72.1°F    PRTN

S STR = ----- D/CM2
```

Figure 10

II.3 Spindle Entry

The user can elect to change the spindle selection by pressing the **SELECT SPDL** key. The **DV-III Ultra** control program will use the previously blank line 3 on the default display screen to record the new spindle input as depicted in Figure 11.

```
RPM:0.0  SPINDLE:31
TEMP:72.1°F    PRTN
ENTER SPINDLE # 31
TORQUE = 0.0 %
```

Figure 11

To enter a spindle number, press the numeric keys until the desired spindle number has been entered. Valid spindle numbers encompass the range from 00 to 99 as listed in **Appendix D**. Mistakes are corrected by repeatedly pressing the numeric keys until the proper spindle value has been entered. At that point, the user presses the **SELECT SPDL** key again. An invalid spindle entry will result in a "**beep**" and the display of the data entry error screen as depicted below.

```
INVALID DATA ENTRY
   "BAD SPINDLE"

  ** RE-ENTER **
```

Figure 12

An invalid spindle entry is any two digit number in the range from 01 to 99 which is not listed in **Appendix D**. This error message will be displayed for a few seconds after which the spindle entry screen (Figure 11) will be re-displayed with a blank field for the spindle number. The user can cancel spindle entry at any time by pressing the **MOTOR ON/OFF/ESCAPE** key.

The user may elect to use a special spindle whose selection is accomplished by first entering a spindle number of **99** and then pressing the **SELECT SPDL** key. This will result in the following display:

```
SPECIAL SPINDLES
1 = AA     4 = AD
2 = AB     5 = AE
3 = AC
```

Figure 13

At this point, press the numeric key for the special spindle of choice. This list is created at the time the Rheometer is manufactured. This list will therefore depend on the number of special spindles ordered and could contain as few as one (1) or as many as five (5) spindles. If no special spindles were purchased, the following message will be displayed if **99** is entered for a spindle number:

```
INVALID DATA ENTRY
"NO SPECIAL SPINDLE"

** RE-ENTER **
```

Figure 14

Press any key to exit this screen and to return to the spindle selection screen. The user may again select another spindle or press the **SELECT SPDL** key to cancel spindle selection operation.

Successful selection of a spindle at the press of the **SELECT SPDL** key returns the user to the default screen with the new spindle displayed in the upper right-hand corner. For standard spindles this would be the two (2) digit designator used to select the spindle. In the case of special spindles, the two (2) letters (**AA, AB, AC, AD** or **AE**) corresponding to the special spindle would be displayed instead. The spindle number or letters will be retained in memory when power is removed. This means that the last value entered for the spindle will be displayed the next time the Rheometer is turned on.

II.4 Direct Speed Entry

At this point, the user may choose to enter a speed by the so-called direct speed entry method. Enter a valid speed in the range of **0.01** to **250 RPM** by pressing the numeric keys successively. The previously blank line 3 on the default display screen records the user's new speed input as depicted in Figure 15:

```
RPM:0.0  SPINDLE:31
TEMP:72.1°F    PRTN
ENTER NEW RPM:11_
TORQUE = 0.0 %
```

Figure 15

Here, the user intends to enter a speed of **112 RPM**, has pressed the "**1**" key twice and is about to press the "**2**" key. If the user makes more than five (**5**) key presses, the **DV-III Ultra** control program will "**roll**" the cursor back to the first character of the field and begin to overwrite the previous data entry.

Next the user presses the **ENTER** key to accept the speed. The motor will begin running at **112 RPM** and the display will be updated to the next screen image:

```
RPM:112  SPINDLE:31
TEMP:72.1°F    PRTN

TORQUE = 56.3 %
```

Figure 16

If the speed entered was not valid the Rheometer will display the following message:

```
 INVALID DATA ENTRY
 "BAD SPEED/TIME"

  ** RE-ENTER **
```

Figure 17

After a few seconds, the display returns to Figure 15 with the speed data field cleared and just the underscore cursor awaiting a new entry.

II.5 External Control

The DV-III Ultra Rheometer can be used in conjunction with Brookfield software, RHEOCALC (V. 2. or higher). Through RHEOCALC, all rheometer functions are controlled by the computer. The DV-III Ultra must be set to the external control mode to allow for proper communication with RHEOCALC. To configure the external control mode, connect cable DVP-80 to the serial port on the DV-III Ultra base **before** turning on the DV-III Ultra. With the DVP-80 cable in place, the DV-III Ultra will present the screen shown in Figure 18 when it is turned on. If external control is selected, the DV-III Ultra will display Figure 19 and only accept control commands from RHEOCALC software.

```
      BROOKFIELD
DV-III ULTRA RHEOMETER
1 = EXTERNAL CONTROL
2 = STANDALONE MODE
```

Figure 18

```
      BROOKFIELD
DV-III ULTRA RHEOMETER
      V0.0  HA
      EXTERNAL
```

Figure 19

The DV-III Ultra may be set to stand alone mode by turning it OFF and ON again and selecting "Stand Alone" or by removing the DVP-80 cable prior to turning the DV-III Ultra on.

Note:	The DV-III Ultra cannot communicate with RHEOLOADER or EZ-Yield software in the external control mode. Chose "Stand Alone" when presented with Figure 18 if you want to use RHEOLOADER.

III. MAKING VISCOSITY AND YIELD MEASUREMENTS

III.1 Quick Start

Viscosity Measurement

The DV-III Ultra Rheometer uses the same methodology for viscosity measurement as the Brookfield Dial Reading Viscometer and DV series of Digital Viscometers. If you have experience with other Brookfield equipment, this section will give you the quick steps for taking a viscosity reading. If you have not used a Brookfield Viscometer before, skip this section and go to Section III.2 for a detailed description.

A) Assemble and level the rheometer (Section I.9).

B) Autozero the rheometer (Section II.1).

C) Enter the spindle number using the SELECT SPINDLE key (Section II.3).

D) Introduce the spindle into the sample and attach the spindle to the coupling nut.
NOTE: Left-hand threads.

E) Enter the speed of rotation using the number pad and ENTER key (Section 11.4).

F) Record % torque and viscosity.

Yield Measurement

The DV-III Ultra Rheometer uses the same methodology for yield measurement as the Brookfield YR-1 Rheometer. If you have experience with the YR-1, this section will give you the quick steps for taking a yield stress reading. If you have not used a Brookfield Viscometer before, skip this section and go to Section IV.6 for a detailed description.

A. Assemble and level the rheometer (Section I.9).

B. Autozero the rheometer (Section II.1).

C. Start the EZ-Yield software in one of the following manners:
   - Click on its associated icon.
   - Click the Windows **START** button. Select **Run**. Enter the name of the program, “ezyield.exe”, (including the path) and click **OK** to execute EZ-Yield.

D. Once EZ-Yield is running, set the COM Port setting on the toolbar to the appropriate COM port.

E. Click on the **Test Parameters** tab beneath the toolbar. Select the appropriate values for all the test parameters listed at the left of the **Test Parameter** page. See Section II.3 for more information regarding how to select **Test Parameters**. Save the test parameters if desired by clicking the **SAVE** button or by clicking **Save** on the **File** menu.

F. Click the **DOWNLOAD** button on the toolbar or click **Download Program** in the **Utility** menu.

G. Ensure the appropriate spindle is attached to the DV-III Ultra and that the sample is properly prepared.

H. Click the down arrow on the **Run** button or click **Run Program** on the **Utility** menu. Select the program number you wish to run - in this case, the program number specified in the list of test parameters.
I. After a few seconds, the **Graphs** page is automatically displayed, signaling that the test has begun. Once all the optional preparation steps are complete, the YR-1 begins sending data to the EZ-Yield software and displaying it on the graph and in the data table on the **Data** page.
J. When the test is complete, a dialog box appears asking you to save the data just acquired.

III.2 Preparations for Making Measurements

A) RHEOMETER: The DV-III Ultra should be turned on, leveled and autozeroed. The level is adjusted using the three feet on the bottom of the base and confirmed using the bubble on the top of the head. Adjust the feet until bubble is inside the center target. Set the level prior to autozero and check the level prior to each measurement.

Proper level is essential for correct operation of the DV-III Ultra.

B) SAMPLE: The fluid to be measured (sample) must be in a container. The standard spindles supplied with the DV-III Ultra (LV(1-4), RV(2-7), or HA/HB(2-7)) are designed to be used with a 600 ml low form Griffin beaker (or equivalent container with a diameter of 8.25cm). The same applies to the optional RV1, HA/HB1, and Vane spindles. Many other spindle systems are supplied from Brookfield with specific sample chambers such as the Small Sample Adapter, UL Adapter and Thermosel.

Brookfield recommends that you use the appropriate container for the selected spindle. You may choose to use an alternate container for convenience, however, this may have an effect on the measured viscosity. The DV-III Ultra is calibrated considering the specified container. Alternate containers will provide results that are repeatable but not "true."

The LV (1-4) and RV (1-7) are designed to be used with the guardleg attached. Measurements made without the guardleg will provide repeatable results but may not provide "true" results.

When comparing data with others, be sure to specify the sample container and presence/ absence of the guardleg.

Many samples must be controlled to a specific temperature for viscosity measurement. When conditioning a sample for temperature, be sure to temperature control the container and spindle as well as the sample.

Please see our publication, "More Solutions to Sticky Problems", for more detail relating to sample preparation.

III.3 Selecting a Spindle/Speed

The DV-III Ultra has the capability of measuring viscosity over an extremely wide range (for example, the RVDV-III+ can measure fluids within the range of 100-40,000,000 cP) (see Appendix B). This range is achieved through the use of several spindles over many speeds. When measuring yield stress with vane spindles, a broad measurement range is also possible through the choice of multiple spindles.

The process of selecting a spindle and speed for an unknown fluid is normally trial and error. **An appropriate selection will result in measurements made between 10-100 on the instrument % torque scale.** Two general rules will help in the trial and error process.

1) Viscosity range is inversely proportional to the size of the spindle.
2) Viscosity range is inversely proportional to the rotational speed.

In other words: to measure high viscosity, choose a small spindle and/or a slow speed. If the chosen spindle/speed results in a reading above 100%, then reduce the speed or choose a smaller spindle.

Experimentation may reveal that several spindle/speed combinations will produce satisfactory results between 10-100%. When this circumstance occurs, any of the spindles may be selected.

Non-Newtonian fluid behavior can result in the measured viscosity and yield stress changing if the spindle and/or speed is changed. See our publication, "More Solutions to Sticky Problems," for more detail.

When viscosity and/or yield stress data must be compared, be sure to use the same test methodology: namely the same instrument, spindle, speed, container, temperature and test time.

III.4 Multiple Data Points

The majority of viscosity and yield stress measurements are made at the quality control level and often consist of a single data point. The test is conducted with one spindle at one speed. The data point is a useful bench mark for the go/no-go decision in a production setting. The DV-III Ultra can be used for single point measurement.

Many fluids exhibit a characteristic change in viscosity and yield stress with a change in applied force. This non-Newtonian flow behavior is commonly seen in paints, coatings and food products as a decrease in viscosity as shear rate increases or an increase in yield stress as rotational speed increases. This behavior cannot be detected or evaluated with the single point measurement.

Non-Newtonian flow is analyzed through the collection of viscosity data over a range of shear rates and the generation of a graph of viscosity versus shear rate (a rheogram). The same applies to yield stress determination by analyzing torque vs. time behavior at different rotational speeds. This information will allow for a more complete characterization of a fluid and may help in formulating and production of the product. The DV-III Ultra is capable of collecting multiple data points for comprehensive analysis of flow behavior. See Section IV on Programming and Analysis.

More information on flow behavior, shear rate and rheograms is available in our publication, "More Solutions to Sticky Problems."

IV. PROGRAMMING THE DV-III ULTRA AND DATA COLLECTION METHODS/ANALYSIS

The programming and data analysis functions of the DV-III Ultra are accessed by pressing the PROG key on the rheometer. The display will change to present a menu with four choices: DV-III, B.E.V.I.S., Models and Yield. DV-III, B.E.V.I.S. and Yield are the programming alternatives. Models will present the five math models available for viscosity data analysis.

```
   PROGRAM MODES
1 = DVIII   3 = MODELS
2 = BEVIS   4 = YIELD
    SELECT:_
```

Figure 20

IV.1 Programming Concepts

The DV-III Ultra may be programmed to collect viscosity or yield data without operator involvement. The captured data may be displayed and analyzed or output to a printer. Programs may be written using three different methodologies: DV-III, B.E.V.I.S and Yield.

The DV-III programming technique uses speed/time pairs, when making viscosity measurements, to control the DV-III Ultra. A program consists of multiple lines (up to 25) instructing the rheometer to operate at a particular speed for some period of time. As an example, we can instruct the DV-III Ultra to rotate the spindle at 5 RPM for 30 seconds and then change speed to 10 RPM and wait 20 seconds with the following program:

Step 1	RPM = 5	Time = 00:30
Step 2	RPM = 10	Time = 00:20

A single data point will be collected at the end of each time interval. Complete details on this programming technique are in Section IV.2.

The B.E.V.I.S. programming technique uses a custom program language to control the DV-III Ultra. A program consists of a series of commands instructing the rheometer in speed control, time control, data collection, temperature control, and output. B.E.V.I.S. offers a higher level of rheometer control compared to the DV-III method. However, the construction of B.E.V.I.S programs is more involved. The 2-step DV-III program previously described is duplicated using B.E.V.I.S. commands below:

```
SSN    5
WTI    00:30
PDN
SSN    10
WTI    00:20
PDN
END
```

The involved programming of B.E.V.I.S. commands is a small trade-off for the significant increase in control capability over the DV-III method. Complete details on this programming technique are in Section IV.3.

The yield programming technique uses three control parameters to run yield tests on the DV-III Ultra: speed control, time control and torque change between successive data points. Complete details on this programming technique are in Section IV.4.

IV.2 DV-III Speed/Time Pair Programs for Making Viscosity Measurements

This programming method allows the operator to control the DV-III Ultra through the variables of speed and time. These speed/time pairs instruct the rheometer to operate at a speed of rotation for a certain period of time. Programs can be created with up to 25 steps. The DV-III Ultra can store up to 10 programs. Upon completion of a program, the data may be viewed on the DV-III Ultra display, analyzed or printed to an attached parallel or serial printer.

Two examples of programs are shown below:

Collect Data Over Time

Step	RPM	Time
1	100	00:12
2	100	00:12
3	100	00:12
4	100	00:12
5	100	00:12

Five viscosity data points will be collected over one minute.

Collect Data At Several Speeds

Step	RPM	Time
1	2.5	01:00
2	5	00:30
3	10	00:30
4	20	00:15
5	50	00:15

Five viscosity data points will be collected at five speeds over 150 seconds.

This program mode is accessed by pressing the program key and selecting number 1; 1 = DV-III. The creation, editing and execution of DV-III programs are described in the following sections.

There are two types of test programs:

1) **Next Speed Set** where the test speeds are programmed, and the operator must signal the DV-III Ultra to change speeds (and therefore take a reading) by pressing the ENTER key.

2) **Prog Speed Set** where the DV-III Ultra will perform the test automatically.

Each step of a program has two variables - speed and hold time. The reading is taken at the end of the hold time interval in a Prog Speed Set or when the ENTER key is pressed in a Next Speed Set.

If the first step hold time interval is 0 seconds, the program is a Next Speed Set type. If the first step interval is 1 second or more, the program is a Prog Speed Set type.

SPEED SET SELECTION AND PROGRAMMING

The **DV-III Ultra** viscometer allows for the retention of a maximum of **10** speed sets with up to **25** discrete speeds per speed set. The program locations are numbered 0 through 9. These speed sets are retained in **EEPROM** memory for those times when the **DV-III Ultra** is not powered up. To access a previously programmed speed set or to enter data for a new speed set, the user presses the "**1**" key when in the display of **Figure 20** and is presented with the screen shown in Figure 21:

```
SPEED SET OPERATIONS
1 = ENTER/EDIT A PGM
2 = CLEAR SPEED SET
3 = USE SPEED SET
```

Figure 21

At this point, the user may Enter/Edit, Clear or Use a stored program (Speed Set). Let's startwith Enter/Edit by pressing the "**1**" key:

```
  SPEED SET USAGE
 IN MEM:012589
  AVAIL:3467
USE SET:_
```

Figure 22

In this example, the user is informed that he has 6 speed sets (0,1,2,5,8,9) pre-programmed in memory and 4 speed sets (3,4,6,7) not programmed and available. Select any one of the ten speed sets by pressing the appropriate numeric key. Pressing the **MOTOR ON/OFF/ESCAPE** key at this point would exit the user to the default **PROGRAM MODES** display (**Figure 20**). For now let's assume that the user wants to program a new speed set by pressing the "**3**" key (the first available program slot).

ENTERING A SPEED SET (PROGRAM)

There are two (2) types of programs available to the user: programs with finite step time intervals and programs with zero (0) step time intervals. We will cover the inputting of finite step time programs first.

SPEED SETS WITH FINITE STEP TIMES (PROG SPEED)

These programs when executed will automatically progress from step to step based on the time intervals programmed by the user. On pressing the "**3**" key in **Figure 22** the user is presented with:

```
 NEW SPEED SET #3
STEP 01
STEP RPM  = 0.0
STEP TIME = 00:05
```

Figure 23

This screen reminds the user of the speed set that he has selected to program and then allows him to change either the speed or time interval or both for that step.

Note: The time interval on entry to this screen will always be set to 00:05 seconds as the default value. The user may of course change it to any <u>valid</u> time of his choice. Whenever you change time interval, that new time becomes the default interval until it is again changed by the user. Also, note that zero (0) times are not allowed for program steps after the first step for **Finite Step** programs.

The **OPTION/TAB** key is used for moving from input field to input field and the **ENTER** key to accept the current input for a step. On entry to this screen, the underscore cursor would be flashing (as shown) under the first digit of the step RPM. Use the numeric keys to make changes to the step speed, repeating the input as many times as required until satisfied.

When satisfied with the speed input, press the **OPTION/TAB** key which moves the flashing cursor down to the first character of the time field. The same procedure is used here to input the step time as was used to enter the RPM above. Speed or time data that is out of range, as defined by **Table 1**, will result in the following screen:

```
INVALID DATA ENTRY
 "BAD SPEED/TIME"

  ** RE-ENTER **
```

Figure 24

This screen will be displayed for **1-3** seconds.

When ready, the user may press the **PROG RUN** key to display the data for the next step in the program, or the **MOTOR ON/OFF/ESCAPE** key (whereby none of the changes up to that point will be accepted) to return to the screen of **Figure 22**. To end a program, the user simply enters and accepts a step RPM and Time of zero (0) or continues to input step data until the program reaches the twenty-five (25) step program limit. In either case, the following screen will be displayed:

```
PROGRAM #3 COMPLETE
1 = ENTER/EDIT A PGM
2 = CLEAR SPEED SET
3 = USE SPEED SET
```

Figure 25

To use the currently selected speed set, press the "**3**" key in **Figure 25**. This would immediately revert to the default screen modified as follows:

```
RPM:0.0  SPINDLE:
TEMP:72.1°F    PRTO
SPEED SET 3 SELECTED
TORQUE = 00.0 %
```

Figure 26

> **Note:** If at this point, prior to using the program the user wished to enter a direct speed, a press of any numeric key which would result in a display similar to **Figure 15**. At the completion of the direct speed input, the display would revert to **Figure 26** above with the appropriate **RPM** displayed, and the viscometer running at that speed.

The program is initiated by pressing the **PROG RUN** key. See "Using Pre-programmed Speeds."

SPEED SETS WITH ZERO STEP TIMES

These programs when executed will require that the user press the **ENTER** key to progress from step to step. On pressing the "**3**" key in **Figure 22** the user is presented with the same screen that he saw in the above description for finite step programs:

```
 NEW SPEED SET #3
STEP 01
STEP RPM  = 0.0
STEP TIME = 00:05
```

Figure 27

The user inputs his step RPM exactly as he did for finite step time programs above. However, for time, input 00:00 and press the **ENTER** key. From this point forward, the user will only be able to enter speeds since each press of the **ENTER** key will advance him to the next step. The **OPTION/TAB** key will not be required. If the user wishes to correct the speed input, continue to press the numeric/ decimal point keys until satisfied. To correct a speed after pressing the **ENTER** key for that step, wait until the program is complete and then edit the program to correct the mistake. To end a program, simply enter and accept a step RPM of zero (0) or continue to input step data until the program reaches the twenty-five (25) step program limit. Speed restrictions/limits are the same as for the description just above as are the error messages.

EDITING A SPEED SET (PROGRAM)

This item is used to review a just-entered program or to review/modify (edit) a program already stored in a memory slot. Entry to this method would typically be from **Figure 22** after selecting an "**IN MEM**" program slot or by pressing the "**1**" key in **Figure 21** having just finished entering a program. In either case, the user is presented with:

```
REVIEW SPEED SET #3
STEP 01 OF 03
STEP RPM  = 10.0
STEP TIME = 00:05
```

Figure 28

Operation in this mode is exactly the same as for entering a new speed set; all key actions and speed and time limits are the same. At this point, the user may continue to review/modify the speeds comprising speed set #**3** or elect to print a listing of the speeds in this speed set. To accomplish this, the user must be in the program Enter/Edit mode; have selected or programmed a speed set which contains more than two (2) speeds, and then press the **PRINT** key. If all is well (i.e. satisfied the above requirements) the rheometer will display:

```
PRINT SPEED SET #3

READY PRINTER THEN
PRESS PRINT KEY.
```

Figure 29

This message simply asks the user to make sure the printer is ready (it's on-line and has paper in it) and then awaits for the **PRINT** key to be pressed. When it is pressed, the **DV-III Ultra** will send the following data to the attached printer:

- **FOR SPEED SETS WITH FINITE STEP TIMES**

Program Use: ____

Programmer:_____________________________

BROOKFIELD DV-III+ RHEOMETER — DATA FOR SPEED SET #9

SPEED #01	RPM = 2.5	TIME INTERVAL = 00:05
SPEED #02	RPM = 5.0	TIME INTERVAL = 00:05
SPEED #03	RPM = 10.0	TIME INTERVAL = 00:05
SPEED #04	RPM = 20.0	TIME INTERVAL = 00:05
SPEED #05	RPM = 50.0	TIME INTERVAL = 00:05

- **FOR SPEED SETS WITH ZERO STEP TIMES**

Program Use: ____

Programmer:_____________________________

BROOKFIELD DV-III+ RHEOMETER — DATA FOR SPEED SET #8

SPEED #01	RPM = 10.0	TIME INTERVAL = 00:00
SPEED #02	RPM = 20.0	TIME INTERVAL = 00:00
SPEED #03	RPM = 30.0	TIME INTERVAL = 00:00
SPEED #04	RPM = 40.0	TIME INTERVAL = 00:00

Since speed sets can contain twenty-five (25) separate speeds, printing the speeds that comprise a speed set will be of great help in allowing the user to fully exercise the power of the **DV-III Ultra**. After the printing is complete, the user will be returned to the display of **Figure 28**.

♦ CLEARING A SPEED SET FROM MEMORY

Since **10** speed sets can be retained in memory, the user may eventually use all the available speed set slots. The user may also have programmed speed sets that are no longer required and would like to remove. Assume that the user had programmed a new speed set #**3** above. If he wished to **<u>permanently</u>** remove that new speed set, or any other **<u>IN MEMORY</u>** speed set he would, while in the screens of **Figure 21**, press the "**2**" key and be presented with:

```
  CLEAR SPEED SET
   IN MEM:0123589
   IN USE:3
CLEAR SET:_
```

Figure 30

This screen advises that there are **7** speed sets in memory; speed set **#3** is in use and that the **DV-III Ultra** is awaiting input for the speed set to delete.

> **Note:** If no speed set is in use the word "**NONE**" will appear next to the **IN USE:** prompt.

At this point, the user has two options:

1. Pressing the **MOTOR ON/OFF/ESCAPE** key will exit from this screen and no speed sets will be cleared. *Or*

2. Pressing any of the keys "**0**", "**1**", "**2**", "**3**", "**5**", "**8**", or "**9**" will delete that speed set.

Thus, to discard speed set #**5**, the user would press the "**5**" key and be presented with:

```
DELETE SPEED SET 5

   ARE YOU SURE?
1 = YES      3 = NO
```

Figure 31

In which the **DV-III Ultra** is requesting that the user specifically press the "**1**" key in order to delete the desired speed set.

```
  CLEAR SPEED SET
   IN MEM:012389
   IN USE:3
CLEAR SET:_
```

Figure 32

Pressing the "**3**" key will cause the **DV-III Ultra** to take no action will return the user to the **CLEAR SPEED SET** opening screen, **Figure 30**. Any attempt to delete an in-use speed ("**3**" for instance) will cause the **DV-III Ultra** to issue a "beep" "beep" with no action being taken. Thus no active (i.e. selected for use) program can be deleted from this screen.

USING PRE-PROGRAMMED SPEEDS

Pressing the three (**3**) key from **Figure 21** takes the user to the speed set selection screen of **Figure 22** where the user selects a new speed set. That done the user is sent to the default screen with: "**SPEED SET X SELECTED**" displayed on line three (3) of the screen.

The user initiates the use of programmed speeds by pressing the **PROG RUN** key. If the user presses the **PROG RUN** key with no speed set selected, the following error box will be displayed:

```
    ** ERROR **
A SPEED SET HAS NOT
BEEN SELECTED
   PRESS ANY KEY
```

Figure 33

However, we will assume at this point that we have selected speed set #**2** for use in the ensuing data gathering operations. To initiate the use of this speed set (with finite step times or with zero step times), the user presses the **PROG RUN** key and is presented with a start/end step input screen as shown next:

```
   SPEED SET #2
TOTAL STEPS = 13
START STEP: 01
  END STEP: 13
```

Figure 34

If the user had not previously entered start and end steps, this screen will display **01** for the start step, and the last program step (**13** in this case) as the end step. The user could elect to use the entire speed set at this point by pressing the **PROG RUN** key. If the user had previously selected a start and end step, those values would be displayed upon entry to this screen instead of the program limit values as shown above. However, while a speed set can contain up to **25** separate speeds, the user may be in a situation where only a few **contiguous** steps may be required. Therefore, this screen allows for the option of entering the range of speeds encompassed by the start step (not necessarily the first step) and the end step (not necessarily the last step). Pressing any numeric key at this point will erase the currently displayed start step and substitute the new value. The user may select a start step less than the end step (a so-called **Up Ramp**) or a start step greater than the end step (a so-called **Down Ramp**). Any attempt to enter a start or end speed not contained in the speed set will result in the following display:

```
INVALID DATA ENTRY
"BAD STEP INPUT"

** RE-ENTER **
```

Figure 35

The **ENTER** key is used to step from the start step entry to the end step entry. Repeated pressing of the **ENTER** key will allow the user to move back-and-forth between the start and end entries and change them as required until the correct start and end step values have been entered. The selected speed set, and the start and end steps values entered, will be retained in EEProm memory for use the next time the viscometer is powered up in the stand-alone mode. Pressing the **OPTION/TAB** key at this point results in the following screen display:

```
LOCKOUT OPTIONS

1 = Enable Lockout
3 = Disable Lockout
```

Figure 36

♦ LOCKOUT OPTIONS

Pressing the "**1**" key **locks out** any use of the NUMERIC keys, and the **PROGRAM**, **SELECT SPINDLE** and **OPTIONS/TAB** keys. Pressing the "**3**" key would disable an existing lockout condition only when in the **LOCKOUT OPTIONS** screen, **Figure 36**. After pressing the "**1**" or "**3**" keys the user would be returned to the display of **Figure 34**. From **Figure 34** the user runs the program by pressing the **PROGRAM RUN** key which signifies that the user is satisfied with his start and stop step values, and wishes to start running with the selected speed set. Or, he can return to the default screen of **Figure 7** by pressing the **MOTOR ON/OFF/ESCAPE** key. (**Note**: the **OPTIONS/TAB** key is re-enabled at this point only to allow the user to return to the **LOCKOUT OPTIONS** screen). In either case, if the user has enabled the lockout mode, the top line of the default display will change as shown in **Figure 37** below:

```
RPM:0.0  LK SPDL:31
TEMP:72.1°F    PRTO
SPEED SET 2 SELECTED
TORQUE = 00.0 %
```

Figure 37

The "**LOCKOUT**" condition will remain in effect until the user re-starts the program mode by pressing the **PROGRAM RUN** key re-initiating the steps of **Figures 34** through **37** above.

Once the start and end steps have been selected, the program is started by pressing the **PROG RUN** key. However, if the viscometer motor was not turned on, the following message would be displayed:

```
MOTOR MUST BE TURNED
ON PRIOR TO RUNNING
PROGRAMMED SPEEDS.
```

Figure 38

At this point, the user would turn the viscometer motor on by pressing the **MOTOR ON/OFF/ ESCAPE** key. This will cause the viscometer to start running at the first selected program speed.

♦ USING PROGRAMMED SPEEDS WITH A ZERO TIME INTERVAL

Assume the following:

- A speed set has been selected.
- A subset of the speed set has not been selected. (i.e. we will use the entire set of speeds)
- The speed set included a time interval that was equal to zero minutes and zero seconds.
- Print mode has been set to non-continuous mode (i.e. output will be sent to the printer only when the user presses the **ENTER** key).

With the above items in effect our default display screen will appear as follows after the **PROGRAM** steps have been completed:

```
RPM:112  SPINDLE:31
TEMP:72.1°F    PRTN
SET 2 01-12@02 ENTER
TORQUE = 56.3 %
```

Figure 39

Or, if the user had selected the **LOCKOUT** option, the default screen above would appear as in **Figure 40** below:

```
RPM:112  LK SPDL:31
TEMP:72.1°F    PRTN
SET 2 01-12@02 ENTER
TORQUE = 56.3 %
```

Figure 40

Here, the **LOCKOUT** mode is indicated by the revision to the top line of **Figure 40** where "**SPINDLE**" has been contracted to "**SPDL**" and the two-letter combo: "**LK**" is being used to indicate the "**LOCKOUT**" condition. Please note that while **TORQUE** is presently being displayed, there is nothing to prevent the user from pressing the **SELECT DISP** key to change the data display item.

The item to note here is the message being displayed on line 3 informing the user that the program is at speed #**02** of the **12** speeds comprising speed set #**02**. The **DV-III Ultra** will continue to operate at **112 RPM** until the **ENTER** key is pressed. At that point (pressing the **ENTER** key), two distinct events will occur:

1. The **DV-III Ultra** will ramp up (or down) to programmed speed #**03** and,
2. Since the printer is **On** (**PRTN**), the data corresponding to the last speed (**112 RPM**) that existed at the time the **ENTER** key was pressed will be sent to the attached printer.

Thus, if **12** speeds comprised the selected speed set, then the user would be required to press the **ENTER** key **12** times to exercise the entire speed set. After the last speed has been executed (i.e. the user pressed the **ENTER** key for the twelfth time), <u>the viscometer speed will be set at **ZERO RPM**</u> and the following message will be displayed:

```
  PROGRAM COMPLETE

    PRESS EITHER
ENTER OR PROGRAM RUN
```

Figure 41

Pressing the **ENTER** key would return the user to the default screen, as depicted in **Figure 40**, or to the program start and stop limit selections of **Figure 34** if the **PROG RUN** key is pressed.

USING PROGRAMMED SPEEDS WITH A NON-ZERO TIME INTERVAL

Assume the following:

- A speed set has been selected.
- A subset of the speed set has <u>not</u> been selected. (i.e. we'll use all of the speeds in the speed set)
- The speed set included a time interval that was greater than zero (0) minutes and zero (0) seconds.
- Print mode has been set to non-continuous mode (i.e. output will be sent to the printer only when the user presses the **ENTER** key).

With the above items in effect our default display screen will appear as follows after the program steps have been completed:

```
RPM:112  LK SPDL:31
TEMP:72.1°F    PRTN
SET 2 01-12@02 PROG
TORQUE = 56.3 %
```

Figure 42

The difference between this mode and the zero time interval mode is reflected in the message being displayed on line three of the display where **PROG** has replaced **ENTER** indicating that the program will be executed step-by-step without user intervention. Here the user is again informed that he is at speed #**02** of the **12** speeds comprising speed set #**02**. As the programmed time interval elapses, the following will occur:

- The **DV-III Ultra** will ramp up (or down) to programmed speed #**03** and,
- The data corresponding to the last speed (speed #**02**) that existed at the moment the step time elapsed will be sent to the printer since the printer is **ON** (**PRTN**) (The automatic ramping to the

next speed will be interpreted by the **DV-III Ultra** as if the **ENTER** key had been pressed, causing the printer output).
- At the completion of the speed set, <u>the viscometer speed will be set at **ZERO RPM**</u>

After the last speed has been executed, <u>the viscometer speed will be set at **ZERO RPM**</u> and the following message will be displayed:

```
 PROGRAM COMPLETE

   PRESS EITHER
ENTER OR PROGRAM RUN
```

Figure 43

Pressing the **ENTER** key would return the user to the default screen, as depicted in its general form in **Figure 42**, or to the program start and stop limit selections of **Figure 34** if the **PROG RUN** key is pressed.

OPERATION WITH PROGRAMMED SUBSET SPEEDS

Had the user selected a subset of a speed set (**Figure 34)**, say speeds #**04** through #**08**, then the programmed mode (with a non-zero time interval) would result in the following display:

```
RPM:112  LK SPDL:31
TEMP:72.1°F    PRTN
SUB 2 04-08@04 PROG
TORQUE = 56.3 %
```

Figure 44

Here, the user is informed that speed set #**2** is being used starting with speed number four and ending at speed #**08** while currently executing speed #**04**. After the first speed (number #**04**) is completed, the display would be updated to show the new speed and the step display would now reflect the current executing step number as in **Figure 45** below.

```
RPM:120  SPINDLE:31
TEMP:72.1°C    PRTO
SUB 2 04-08 @05 PROG
TORQUE = 72.8 %
```

Figure 45

If the speed set contained <u>**no**</u> time interval, the selection of a subset of speeds would result in a screen display as shown in **Figure 46**.

```
RPM:112  LK SPDL:31
TEMP:72.1°F    PRTN
SUB 2 04-08@04 ENTER
TORQUE = 56.3 %
```

Figure 46

The step number would be incremented each time the user pressed the **ENTER** key. Assume that the speed corresponding to step #**04** was executing. If the user presses the **ENTER** key, our display will be updated as shown in **Figure 47**

```
RPM:120  LK SPDL:31
TEMP:72.1°F    PRTN
SUB 2 04-08@05 ENTER
TORQUE = 72.8 %
```

Figure 47

♦ PROGRAMMED SPEED STOP

The user may stop program mode operation at any time by pressing the **MOTOR ON/OFF/ESCAPE** key anytime during program operation. The following message would be displayed:

```
PROGRAM STOPPED

PRESS ANY KEY
```

Figure 48

The viscometer motor would be automatically turned **OFF** and the default screen display (**Figure 37**) would show zero (0.0) **RPM**. At this point, the user may perform any valid viscometer operation - load a new speed set; run a direct speed; set alarms or even **re-start** the current program.

♦ PROGRAMMED SPEED HOLD

The user may interrupt program mode operation at any time by pressing the **PROG** key during program execution. The following message would be displayed:

```
PROGRAM HOLD

1 = CONTINUE PROGRAM
3 = STOP PROGRAM
```

Figure 49

The viscometer motor would be automatically turned **OFF** and the **DV-III+** would be awaiting user key input. If the user presses the “**1**” key, the **DV-III Ultra** will continue executing the current program, picking up from whichever **RPM** (and time, if applicable) it had been interrupted. However, if the user had pressed the “**3**” key, operation would be identical to that described above for **Programmed Speed Stop** mode**.**

IV.3 Bevis Programs for Making Viscosity Measurements

The B.E.V.I.S. Programming Method allows the operator to control the DV-III Ultra through the variables of speed, temperature and time while providing for independent data collection. Programs can include up to 25 commands with a maximum data count of 800. The DV-III Ultra can store up to 10 programs. Upon completion of the program the data may be viewed on the DV-III Ultra display, analyzed or printed to an attached parallel or serial printer.

B.E.V.I.S. programs are created on a PC using Rheoloader software (supplied with the DV-III Ultra). The programs are "loaded" onto the DV-III Ultra. Loaded programs cannot be deleted, but can be overwritten.

IV.3.1 Setting Up Bevis Programs

B.E.V.I.S. (Brookfield Engineering Viscometers Instruction Set) is a scripting language developed at Brookfield Engineering Laboratories that allows for the creation of flexible programs to control our line of Rheometers. In the case of the DV-III Ultra Rheometer, programs are created then loaded into the Rheometer using the RheoLoader software.

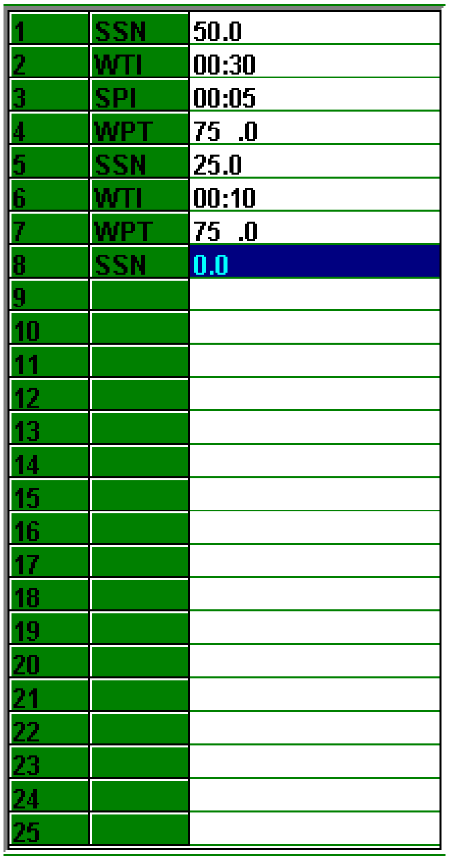
Some features of the scripting language are:
- Repeatedly run the same program for quality control purposes.
- Wait for a prevailing condition before continuing with the program (i.e. torque value, a temperature value, a key press, etc.).
- Run the Rheometer at any of the speeds in the Custom Speed menu.
- Display messages to the screen or an attached printer to aid in operator usability.
- An internal clock that keeps time between each printed data line (this time is displayed as the last parameter on each printed line) providing a consistent time base for the collected data.

IV.3.1.1 Description of B.E.V.I.S. Commands

Command Code	Required Parameter	Command Description
WTI	Time (MM:SS)	The program waits at this step until the specified time elapses.
WPT	% Torque value (%)	The program waits at this step until the current % torque equals the especifed value.
WTP	Temperature value (°C)	The program waits at this step until the current temperature equals the specified value.
WKY	16 character (or less) text message	The specified message is displayed on the top line of the DV-III Ultra dis play while **PRESS A KEY** is displayed on the bottom line of the DV-III Ultra. The program waits at this step until a viscometer key is pressed. While waiting at this step, the viscometer produces a beep every few second to remind the operator that a keypress is required to continue. If a print interval was enabled (see SPI) at the time this command is executed, the data print timer continues to count up. If the print interval elapses and a key has not yet been pressed, a line of data displaying the time since the last data print is printed as soon as a key is pressed.
SSN	Speed value (RPM)	The DV-III Ultra begins rotating at the specified speed. This can be any of the speeds listed in the Speed list of the DVLoader software. These speeds are the same as those listed in the Custom Speeds list in the viscometer's Options menu.
SPI	Time (MM:SS)	The DV-III Ultra begins printing data to the selected printer (serial or parallel; as selected in the DV-III Ultra menus) at the rate specified. MM:SS is minutes:seconds.
SSP	Two digit spindle code	Calculations of viscosity, shear stress, and shear rate are performed based on the specified spindle code. This command overrides the spindle currently entered via the keypad on the DV-III Ultra.
STM	Temperature value	Set and control to the specified temperature if a Brookfield Engineering Labs. temperature controller is attached to the rheometer.
STZ	N/A	Sets the data print timer clock back to zero.
PDN	N/A	The DV-III Ultra immediately prints a data string to the selected printer (serial or parallel; as selected in the DV-III Ultra menus).
PLN	16 character (or less) text message	The DV-III Ultra prints the specified message to the selected printer (serial or parallel; as selected in the DV-III+ menus).

NOTE: MM:SS is minutes:seconds

By using various combinations of the above commands, programs are created that automatically control the viscometer and collect data (via an attached printer) from the DV-III Ultra Rheometer.

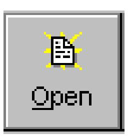


The RheoLoader software is a Windows 95 (or above) based program used to create, save, print and downlaod B.E.V.I.S. programs to the DV-III Ultra Rheometer. Start the software by clicking on its associated icon or by clicking the Start button; select Run; enter the name of the progrram to execute {Rheoad.exe}; then click OK.

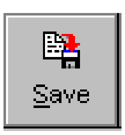
This is the grid where the operator programs are created. It is used to view and edit the B.E.V.I.S. programs. When the software starts, an empty grid appears on the left of the screen. Highlight a command in the list box to the right of this grid, then click on the Insert button to insert the command into the highlighted line of the grid. This same insertion task can also be accomplished by double-clicking on the appropri-ate command in the list box to the right.

Before being permitted to insert another command, the pa-rameter for the previous command in the grid must be entered if one is required. This parameter is entered into the last column of the grid.

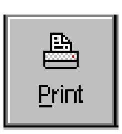
Select the COM (RS-232) port the Programmable DV-III Ultra Rheometer is con-nected to from the COM Port drop down list.



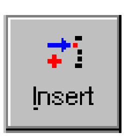
Click the Open File button to load existing B.E.V.I.S. programs.



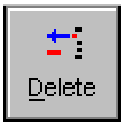
Click the Save File button to save the B.E.V.I.S. program displayed in the grid.



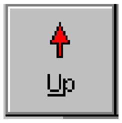
Click the Print button to print the B.E.V.I.S. program displayed in the grid.



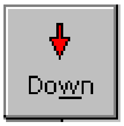
Click the Insert button to insert the B.E.V.I.S. command selected in the Commands box into the selected row in the program grid.



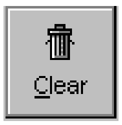
Click the Delete button to delete the command in the selected row of the program grid.



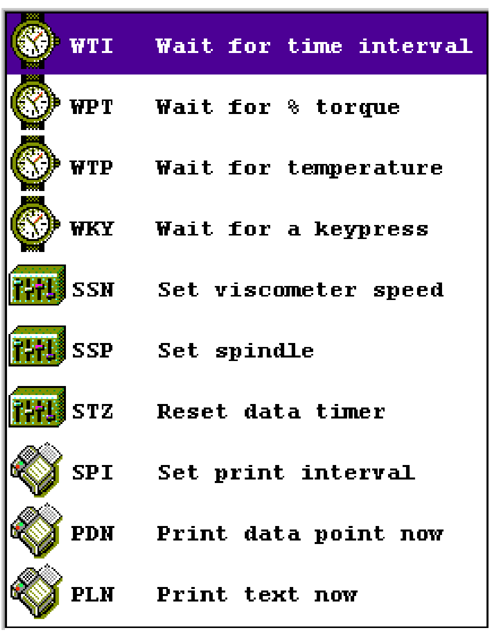
Click the Up button to move the command in the selected row of the program grid up one row.



Click the Down button to move the command in the selected row of the program grid down one row.

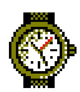


Click the Clear button to clear the grid of all commands. Once cleared, the commands cannot be retrieved.

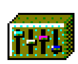


This list box displayed the commands available for creating programs. As previously stated, clicking on the Insert button inserts the highlighted command (WTI in this case) into the selected line in the program grid. Double-clicking on a line in this list box also inserts the command into the grid.

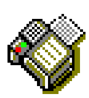
The icons to the left of the command descriptions indicate the type of command:



A command to wait for a condition.



A command to set a program parameter.



A command to send information to an attached printer.

Before downloading a program to the Rheometer, ensure the following have been checked:

- The appropriate cable (BEL Part No. DVP-80) is connected between the selected COM port of the PC and the Rheometer.
- The DV-III Ultra is at the download screen: **OPTIONS/DOWNLOAD A PROGRAM/LOAD TO SLOT#x** where x is slot 1,2,3, or 4.
- With the **LOAD TO SLOT #x** screen displayed, choose a store slot using the DV-III Ultra arrow keys, then press the **ENTER/AUTORANGE** key on the Rheometer. If after five seconds, the Rheometer cannot communicate with the RheoLoader program, the B.E.V.I.S. ERROR NO PC ATTACHED message is displayed. If a connection is established, the Download button on the PC software becomes enabled, and the DV-III Ultra screen displays DOWNLOAD-

ING B.E.V.I.S. PROGRAM TO SLOT #1. Click on this button to download the displayed program to the DV-III Ultra. When the download is complete, the DV-III Ultra displays DOWNLOAD DONE TO EXIT PRESS A KEY.

At this point, the program in the DV-III Ultra can be printed and/or run from the Rheometer.

Click on this button to exit the RheoLoader software.

IV.3.1.2 Example Programs

The following example programs can also be found on the RheoLoader disk that was included with the DV-III Ultra Rheometer:

Program 1: Pre-shear

Command	Command Description	Parameter	Comments
PLN	Print text now	Preshearing now	print user message
SSN	Set viscometer speed	50.0	run at 50 RPM
WPT	Wait for % torque	90.0	wait unitl 90% torque is reached
PLN	Print text now	Collecting data	print user message
SPI	Set print interval	00:10	begin printing data at 10 second intervals
SSN	Set viscometer speed	10.0	run at 10 RPM
WTI	Wait for time interval	01:40	wait at this step for 1 minute and 40 seconds, effectively printing 10 data lines

Program 2: For use with an external temperature controller

Command	Command Description	Parameter	Comments
STM	Set temperature	40.0	Set control value to 40°C
WTP	Wait for temperature	40.0	wait until temperature = 40°C (as an example, a Thermosel/Controller can be used for temperature control)
WTI	Wait for time interval	05:00	soak time; allow temperature to settle
SSN	Set viscometer speed	25.0	run at 25 RPM
SPI	Set print interval	00:30	begin printing data at 30 second intervals
WTI	Wait for time interval	06:00	wait at this step for 6 minutes, effectively printing 12 data lines

Program 3: Wait for cure

Command	Command Description	Parameter	Comments
SSP	Set spindle	31	set to a number 31 spindle
SSN	Set viscomter speed	100.0	run at 100 RPM
SPI	Set print interval	00:05	begin printing data at 5 second intervals
WPT	Wait for % torque	85.0	wait until % torque = 85; a curing cycle

Program 4: Spring relax

Command	Command Description	Parameter	Comments
WKY	Wait for a key press	Wind to 100%	tell operator to wind spindle until 100% torque is reached
WPT	Wait for % torque	100.0	wait until 100% torque is reached
WKY	Wait for a key press	Release spindle	tell operator to release the spindle
SPI	Set print interval	00:01	begin printing data at 1 second intervals
WPT	Wait for % torque	0.0	wait for spindle to completely unwind to 0% torque

Program 5: Variable speed

Command	Command Description	Parameter	Comments
SSN	Set viscometer speed	5.0	run at 50 RPM
WTI	Wait for time interval	00:10	wait for 10 seconds
PDN	Print data point now		print one data point
SSN	Set viscometer speed	10	run at 10 RPM
WTI	Wait for time interval	00:10	wait for 10 seconds
PDN	Print data point now		print one data point
SSN	Set viscometer speed	20	run at 20 RPM
WTI	Wait for time interval	00:10	wait for 10 seconds
PDN	Print data point now		print one data point
SSN	Set viscometer speed	5.0	run at 50 RPM
WTI	Wait for time interval	00:10	wait for 10 seconds
PDN	Print data point now		print one data point

IV.3.2 Running Bevis Programs in Standalone Mode

The B.E.V.I.S. program menu is accessed by pressing the **PROG** key and selecting number 2, 2 = B.E.V.I.S. The loading and execution of B.E.V.I.S. programs are described in the following sections.

B.E.V.I.S. PROGRAMS MENU

B.E.V.I.S. operations are accessed by pressing the "**2**" key when in the **PROGRAM MODES** menu. The user is immediately presented with:

```
B.E.V.I.S. PROGRAMS
PROGRAM #3 IS IN USE
1 = SELECT/DOWNLOAD
2 = RUN
```

Figure 50

This screen informs the user that the **B.E.V.I.S.** program in storage slot **3** is current ("last used") and that it may be run by pressing the "**2**" key or another program may be selected by pressing the "**2**" key. It should be noted that entrance to the **B.E.V.I.S.** program mode makes the last used program available for printing or running. Thus the user, seeing that a program was resident in slot **3**, could have printed it directly from the above screen by pressing the front panel **PRINT** key.

SELECTING AND DOWNLOADING B.E.V.I.S. PROGRAMS

To select a program the user presses the "**1**" key and is presented with:

```
 SELECT A PROGRAM
AVAILABLE:56789
IN MEMORY:01234
  CURRENT:3
```

Figure 51

The user is informed that the current (or "last used") program is no. **3**; that programs **0**,**1,2,3** and **4** are available for immediate use and that five (5) slots: **5,6,7,8** and **9** are empty and are available for download from a host computer. A press of the **ENTER** key would select the current program (i.e. "**3**") for use while a press of an appropriate numeric key **0**,**1,2,3** and **4** would select that specific program slot for use. The above screen would be updated to reflect the new selection. A press of the **5,6,7,8** or **9** keys will place the user in the **B.E.V.I.S.** program download mode. The following screen appears (assuming a press of the "**5**" key):

```
DOWNLOAD A PROGRAM

LOAD PGM TO SLOT #5
PRESS ENTER TO START
```

Figure 52

The user's selection "5" is flashing and is the current slot selection. When ready, the user presses the **ENTER** key to begin the program download. The following screen will appear for the duration of the download. See Section VI. RHEOLOADER for information on creating and downloading B.E.V.I.S. programs.

```
DOWNLOAD A PROGRAM

 DOWNLOADING BEVIS
PROGRAM TO SLOT #2
```

Figure 53

♦ RUNNING B.E.V.I.S. PROGRAMS

Programs are run by pressing the "**2**" key when in **Figure 50** which presents the user with the following screen:

```
B.E.V.I.S. PROGRAMS

  RUN PROGRAM #3
PRESS ENTER TO START
```

Figure 54

Here we see that our program is no. "**3**" and that a press of the **ENTER** key will start it running. As soon as the program starts executing the user will be presented with:

```
RUNNING PROGRAM #3
THIS STEP: WTI0330
NEXT STEP: PDN
STEP 02/39    01:00
```

Figure 55

This is a typical display for a given program step. The current step being executed (**WTI0330**) is shown as well as the next step to be executed (**PDN**). The bottom line displays the current program step, the total number of steps (**02/39**) and any time intervals if they are relevant. The **WKY** command message could be displayed on the bottom line in lieu of the step and time info. This screen stays resident until the user presses the **OPTIONS/TAB** key which "toggles" back-and-forth between this screen and an amended default screen shown next:

```
RPM:0.0  SPINDLE:31
TEMP:72.1°C    PRTN
BEVIS PROG RUNNING
TORQUE = 00.0 %
```

Figure 56

The user now sees viscosity data and can use the **SELECT DISPLAY** key to view other viscosity measurement parameters. Pressing the **OPTIONS/TAB** key from now on will toggle between the screens of **Figure 55** and **Figure 56**. The program code will automatically switch back to the program progress screen, **Figure 55** above, if a conditional has been reached, the end of a program step is reached or user input is required. If no user input is required, the **B.E.V.I.S.** program proceeds to the next step without switching back to the progress screen.

IV.4 Choosing the Best Data Collection Method for Viscosity Tests

The DV-III Ultra offers 3 methods for data collection; Single Speed, DV-III Speed/Time Pairs and B.E.V.I.S. Programs. The decision of which technique is best should be made considering the test requirements.

- **SINGLE SPEED**

  Single speed measurements may be made by direct speed commands on the DV-III Ultra keypad. Viscosity and % Torque are read directly from the display. This technique offers the simplicity of the Brookfield Dial Viscometer. Multiple data points may be gathered by issuing multiple speed commands.

  This technique is fast and easy. It's well suited to gathering data on samples prior to establishing a test method or for performing single point tests.

- **DV-III SPEED/TIME PAIRS**

  DV-III speed/time pairs offers a simple technique to collect multiple data points. Programs can be created and executed from the DV-III Ultra keypad. Results can be analyzed or output to a printer.

  This technique is useful when multiple data points are required and the test method is simple.

- **B.E.V.I.S. PROGRAMS**

  B.E.V.I.S. Programs offer a command set capable of sophisticated rheometer control and data collection. Programs are created on a PC and executed from the DV-III Ultra keypad. Results can be analyzed or output to a printer.

  The B.E.V.I.S. Program technique is useful when sophisticated data collection is necessary.

IV.5 Data Analysis for Viscosity Tests

Data collected from DV-III speed/time pairs or B.E.V.I.S. programs may be analyzed using several math models. These models provide a means to numerically describe the behavior of the test fluid. In the case of viscosity measurement, a non-Newtonian fluid will produce a curve when test data is plotted on a shear stress vs. shear rate graph. The math model will force the data into a straight line and describe it with a slope and y intercept. The terminology associated with the slope and y intercept vary from model to model as does the interpretation of results.

The DV-III Ultra does not allow for data sets to be edited. Programs must be constructed to conform with the following data requirements if math models are to be used:

- The data set must contain non zero values for shear stress and shear rate (except for the paste model which requires non zero viscosity and RPM).
- There cannot be two equal adjacent shear rate values (RPM values for paste model).
- % torque values of all data points must be between 0.1% and 100%.

  If any of the above circumstances are violated, an error message will appear when a math model is selected.

ERROR #1: A % torque value is less than 0.1. A shear stress or shear rate value is zero.

ERROR #2: A % torque value is greater than 100.

ERROR #3: Reserved

ERROR #4: Two adjacent speeds of equal value.

Math models for data analysis are accessed by pressing the PROG key and then 3; 3 = Models (Figure 20). If no data (i.e. no data at all or less than two(2) data points) is in the data buffer, no modeling can be performed and Figure 57 will be displayed:

```
      ERROR
YOU CANNOT EXECUTE A
MATH MODEL BECAUSE
NO DATA IS IN BUFFER
```

Figure 57

If there is already data in the data buffer, the user will be presented with the following screen:

```
    MATH MODELS
1 CASSON    4 PWR LAW
2 NCA/CMA   5 PASTE
3 BINGHAM
```

Figure 58

The user is informed that there are five (5) math models which can be used on the buffer data. A model is selected by pressing the appropriate numeric key. No matter the model selected, the following screen will be displayed for the duration of the mathematical analysis:

```
MATH MODEL

CALCULATING
```

Figure 59

When the calculations are complete, the results for the particular model will be displayed as follows:

```
CASSON (STANDARD) cP
PLASTIC VISC: 3069
YIELD STRESS: 140.1
 CONF OF FIT: 97.5
```

Figure 60

This screen, for the Standard CASSON Model, is typical for all five (5) of the math models. Note the cP in the upper right-hand corner to remind the user that the values are **cP** based. It could have been **SI** (if the user had opted for **SI** display in the **SETUP** menu) as shown on the next two (2) of the following four (4) math model screens:

The user may elect to print test results in order to obtain hard copy results. This is accomplished by pressing the **PRINT** key while any of the above screens are being displayed causing the following to be printed:

♦ STANDARD CASSON PRINTOUT

Sample Name: ____

Operator Name: _________________________

Date: 01/14/1999 Time: 02:27 Math Model Results: CASSON (STANDARD)

Model: HB Spindle: 34

Plastic Viscosity:	1906.3	cP
Yield Stress:	1976.88	D/CM2
Confidence of Fit:	63.6	%

The equation for each model is described below with a definition of parameters. Please contact Brookfield or an authorized representative if further information is required.

> **Note:** The **confidence of fit** parameter used in all of the models is an indication of how well the model fits the data set. 100% indicates the best fit.

1. Casson (Standard)

The Standard Casson equation is: $\sqrt{\tau} = \sqrt{\tau o} + \sqrt{\eta D}$ *where:*

τ = Shear Stress
τo = Yield Stress (stress at zero shear rate)
η = Plastic Viscosity
D = Shear Rate

The calculated parameters for this model are:

Plastic Viscosity	(cP or mPa•s)
Yield Stress	(Dynes/cm2 or N/m2)
Confidence of Fit	(%)

The Standard Casson method is a direct implementation of the original Casson equation.

2. NCA/CMA Casson (Chocolate)

This Casson method is derived from the standard set forth by the National Confectioners Association (NCA) and the Chocolate Manufacturers Assocation (CMA). Although based on the original Casson equation, this implementation has been tailored by the NCA and CMA specifically to applications involving chocolate.

The Chocolate Casson equation is: $(1{+}a)\sqrt{\tau} = 2\sqrt{\tau o} + (1{+}a)\sqrt{\eta D}$ *where*:

τ = Shear Stress
τo = Yield Stress (stress at zero shear rate)
η = Plastic Viscosity
D = Shear Rate
a = spindle (or bob) radius/ inner cup radius

The calculated parameters for this model are:

Plastic Viscosity	(cP or mPa•s)
Yield Stress	(Dynes/cm2 or N/m2)
Confidence of Fit	(%)

3. Bingham Plastic

The Bingham equation is: $\tau = \tau o + \eta D$ *where*:

τ = Shear Stress
τo = Yield Stress (stress at zero shear rate)
η = Plastic Viscosity
D = Shear Rate

The calculated parameters for this model are:

Plastic Viscosity	(cP or mPa•s)
Yield Stress	(Dynes/cm2 or N/m2)
Confidence of Fit	(%)

4. Power Law

The Power Law equation is $\tau = kD^n$ *where*:

τ = Shear Stress
D = Yield Stress (stress at zero shear rate)
k = Plastic Viscosity
n = Shear Rate

The calculated parameters for this model are:
Flow Index (no units)
Consistency Index (cP or mPa•s)
Confidence of Fit (%)

5. IPC Paste Analysis

This method is intended to calculate the Shear Sensitivity Factor and the 10 RPM Viscosity value of pastes. A prime example of its use is in the solder paste industry, thus the name IPC (Institute for Interconnecting and Packaging Electronic Circuits).

The Paste equation is: $\eta = kR^n$ where:
η = Viscosity (cP)
k = Consistency Multiplier
R = Rotational Speed (RPM)
n = Shear Sensitivity Factor

The calculated parameters for this model are:

Shear Sensitivity Factor	(no units)
10 RPM Viscosity	(cP or mPa•s)
Confidence of Fit	(%)

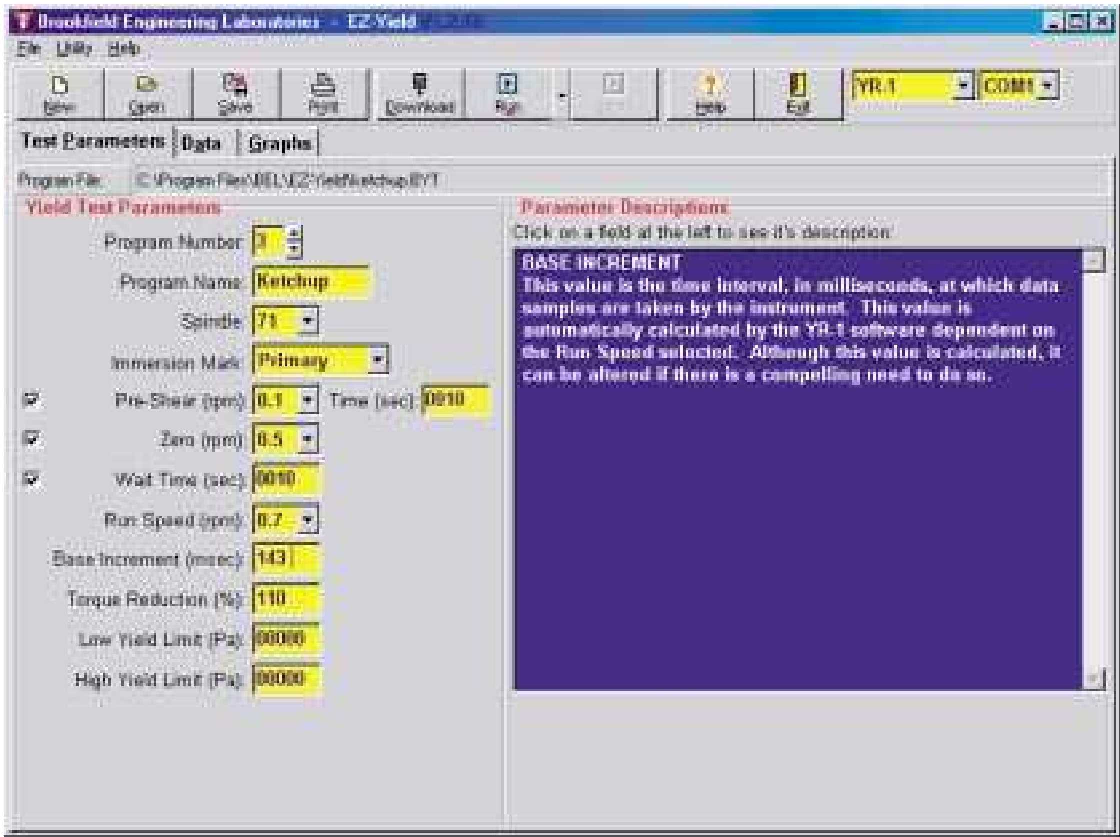
IV.6 Yield Stress Test Programs and Test Examples

This section explains how to create a yield test program for the DV-III Ultra Rheometer. There are no yield test programs stored in instrument memory when the DV-III Ultra is shipped from Brookfield. Therefore, you will need to use the EZ-Yield Software on your PC to create yield test programs. Once a yield test program has been downloaded from your PC to the DV-III Ultra, you will be able to run tests by following the instructions in this section.

Brookfield EZ-Yield software is a 32-bit Windows program supplied with every DV-III Ultra Yield Rheometer.

IV.6.1 Test Parameter Description

When running the EZ-Yield application on your PC, the main screen for creating yield test programs is shown here:



Use this page to create, load, save, and print test parameters that are then downloaded into one of the memory locations in the DV-III Ultra Rheometer. The file name (including path) of the test parameters currently loaded is displayed at the top of the page.

Click on the entry field to edit the information in that field. The fields with downward pointing arrows on the right require that you click that arrow to make a selection from the drop down list. When a field is selected, the text on the right of the page, in the Parameter Descriptions box, changes to give information appropriate to the field selected.

The **Pre-Shear**, **Zero**, and **Wait Time** steps are optional. Clicking the check box to the left of these fields until the check mark appears enables these parameters. Clicking the check box to the left of these fields until the check mark disappears disables these parameters.

Clicking the **NEW** button (or selecting *New* from the **File** menu) sets all parameters to their default values.

All open, save, and print operations selected while this page is displayed will open, save, and print test parameters, but NOT data.

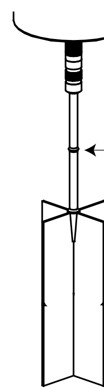
The following is a detailed description of the test parameters involved in creating a yield stress test.

Program Number
This is the number of the memory slot in the DV-III Ultra Rheometer to which the test parameters will be loaded. There are ten (10) slots, numbered from 0 through 9.

Program Name
This descriptive user-supplied name is loaded into the memory slot in the DV-III Ultra Rheometer with the test parameters. A maximum of ten (10) alphanumeric characters may be used for this name.

Spindle
<u>Spindle Number</u>

A two (2) digit code representing the spindle number used for the test must be selected. See Appendix A for more information regarding spindles for use with the DV-III Ultra Rheometer. Selection of the appropriate spindle code is important to ensure correct stress calculations.



<u>Immersion Mark</u>

Each vane spindle has two (2) immersion marks. The primary immersion mark is located on the spindle shaft. Normally, the spindle should be inserted so that the sample reaches this mark. If the sample container is too small to allow the spindle to be inserted to this mark, the secondary immersion mark may be used. This mark appears half way down the blades of the vane spindles. See Appendix D2 for more information regarding spindles and immersion marks.

Note: Selection of the appropriate immersion mark is important to ensure correct stress calculations.

Pre-Shear Information
An optional Pre-Shear step can be included in the test parameters. If the Pre-Shear box is checked on the Test Parameter page of the EZ-Yield software, a pre-shear step will be performed before running the actual test. The user must supply the speed and duration of the pre-shear step. See Appendix B for a complete list of pre-shear speeds.

Pre-shearing is the shearing of sample before measuring its yield properties. This process breaks down the sample's structure. It is particularly useful if the investigator wants to eliminate previous shear history (e.g., bumping, transferring) of the sample before testing and observe the structural rebuilding of the sample. This may simulate the following: ketchup pumped out of a bottle will rebuild after it comes to rest on the French Fries. There are materials whose measured yield stress will be lower after pre-shearing than if tested without pre-shearing. This may be used to compare the rate at which different materials rebuild. The yield stress measured in a pre-sheared sample is the "dynamic yield", while the yield stress measured for an originally undisturbed material is the "static yield".

Zeroing after pre-shearing is performed on the sample is highly recommended for every test. The next section explains how to do this.

Zero

An optional, but highly recommended, torque Zero step can be included in the test parameters. If the Zero box is checked on the Test Parameter page of the EZ-Yield software, the rheometer will rotate the motor in the appropriate direction until 0% torque is achieved.

This may be necessary because the spindle sometimes twists a small amount during insertion into the sample. This results in a small, although possibly significant, torque applied to the sample. Slow zeroing speeds are used to eliminate this initial torque and minimize effects on the sample's structure before the test is started.

Zeroing is an essential step after preshearing.

This gives a consistent starting point for each test. A speed for the step must be supplied in the appropriate box. Faster speeds achieve a “zero” quicker but may cause a more variable starting point. See Appendix D2 for a complete list of zero step speeds.

Wait Time

An optional Wait step can be included in the test parameters. If the Wait Time box is checked on the Test Parameters page of the EZ-Yield software, there will be a time delay after zeroing before the actual test run begins. During this delay, the motor shaft will be at zero (0) RPM.

Wait Time is the time the sample is allowed to rest between the completion of zeroing and the start of the yield measurement. Some samples rebuild structure more slowly than others after shearing, such as during handling, pouring sample into a beaker, etc. Certain low-viscosity paints may also have a low yield stress. Waiting 30 seconds, for example, after immersing the spindle may allow the sample to rebuild, producing a more consistent test method.

Run Speed

Run Speed is the motor speed for the DV-III Ultra at which the material is tested. It is common for materials to appear stiffer when tested at higher speeds. That is, the slope of the stress-vs.-strain curve increases with increasing speed. This is because the material structure has less time in which to react to dissipate the applied stress. Increasing the speed will, in most cases, increase the yield stress measured by the instrument. Most yield tests are conducted at relatively low speeds (<1 rpm) to minimize any inertial effects when using vane spindles. See Appendix D2 for a complete list of run speeds.

Base Increment

Base Increment is the amount of time in milliseconds between data points used for taking torque (stress) readings. The software automatically calculates base increment values. Smaller base increment values are used during faster speed tests to ensure that data are taken fast enough to properly determine the yield point. Larger base increment values are used during slower speed tests because these tests are expected to take longer and the longer time between data points helps prevent typical data files from becoming very large. However, the user may still wish to set this value to suit the required need after some familiarity is gained with the material being tested.

The % torque data are used to determine when a yield point is reached. The rigid line incremental torque value is automatically calculated by the EZ-Yield software and depends on the Run Speed selected. Although this value is calculated, it can be altered if there is a compelling need to do so. If the Base Increment has been manually altered for any reason, selecting a new Run Speed automatically resets the Base Increment to its optimum value.

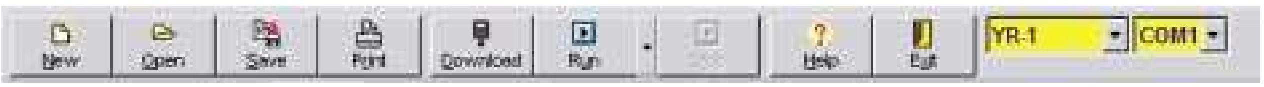
Torque Reduction

Torque Reduction is percentage change in torque value between successive data points. That is, the material yields or begins to break down and, as a result, the measured incremental torque begins to decrease. A value of 100% for this parameter causes the test to stop as soon as there are no torque increases during a base time increment. Some users may wish to see a drop in torque after the yield point. Setting this parameter to values greater than 100% allows data to be collected after the yield point by the EZ-Yield™ software so the decrease in torque may be more easily visualized. However, this will also affect the calculated yield stress value.

Low and High Yield Limits

These values (in Pa) can be used as a Quality Control tool. If the resulting yield stress from a test falls outside these limits, an appropriate message is displayed and printed with the results. Entering zero (0) for both these parameters disables this QC feature.

IV.6.2 Toolbar and Menu Options



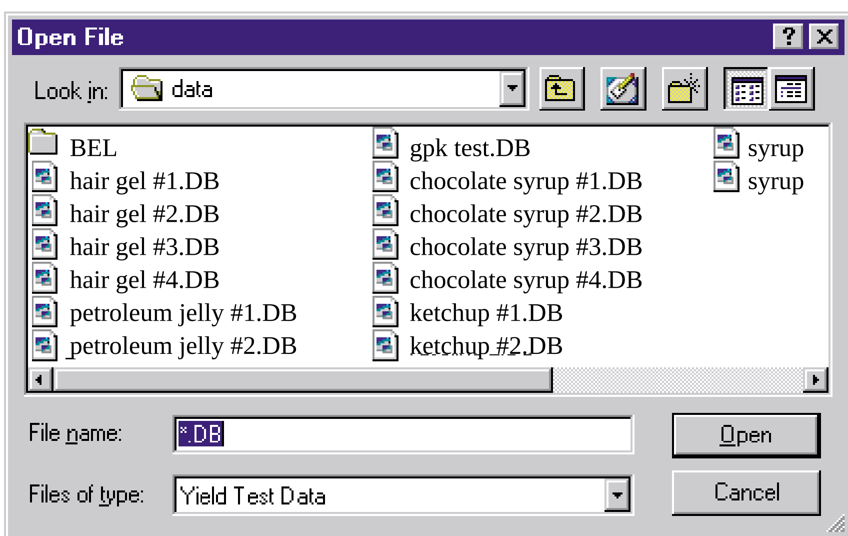
Note: All functions shown on the toolbar can also be accessed from the menu above the toolbar.

New

Selecting **New** (using the **New** button or the **New** option in the **File** menu) displays the Test Parameters page with default values in each field. Use this function before creating a new test program.

Open

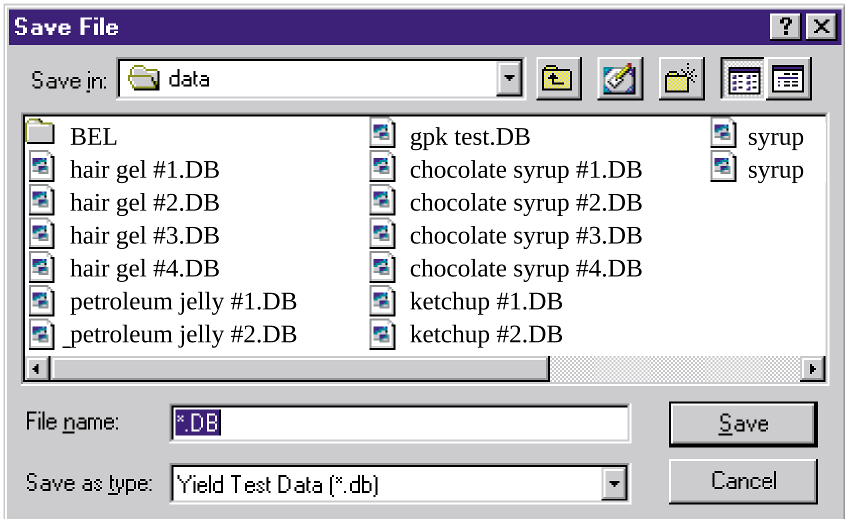
Selecting **Open** (using the **OPEN** button or the **Open** option in the **File** menu) displays the **Open File** dialog box.



The **Open File** dialog box automatically displays files of the type it thinks the user requires; i.e., if the **Test Parameters** page was displayed, the file types displayed are test parameter files (*.BYT) and if the **Data** or **Graph** pages were displayed, the file types displayed are data files (*.DB). Regardless of the file types initially displayed, either type of file can be loaded by selecting the desired type at the bottom of the **Open File** dialog using the **Files of Type** drop down list.

Save

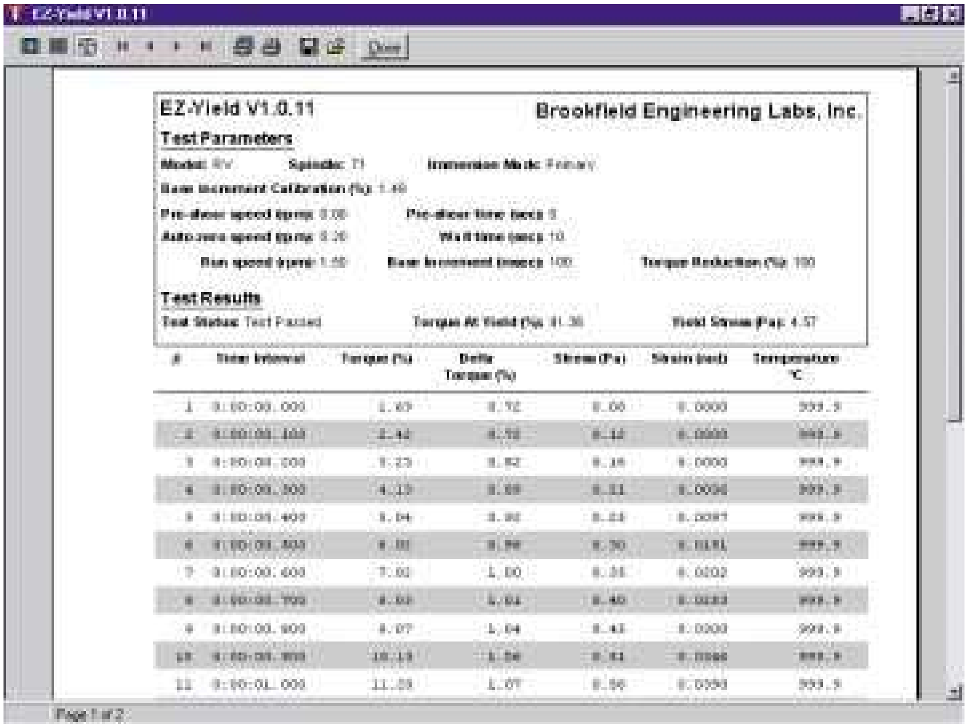
Selecting **Save** (using the **SAVE** button or the **Save** option in the **File** menu) displays the **Save File** dialog box.



The **Save File** dialog box automatically displays files of the type it thinks the user requires (i.e. If the **Test Parameters** page was displayed, the file types displayed are test parameter files (*.BYT) and if the **Data** or **Graph** pages were displayed, the file types displayed are data files.). Regardless of the file types initially displayed, either type of file can be saved by selecting the desired type at the bottom of the **Save File** dialog using the **Files of Type** drop down list.

Print

Selecting **Print** (using the **PRINT** button or the **Print** option in the **File** menu) displays a **Print Preview** window. Once this window is displayed, clicking the **PRINT** button sends the report to the selected printer. The functions of the buttons on the toolbar are described below:



Zoom To Fit — Displays a complete page of the report in the window. Text and graphics are shrunk so that a complete page will fit on the screen.

Zoom To 100% — Increases the size of all text and graphics so that they are displayed at 100% of their actual size.

Zoom To Width — Displays a page so that the complete width of the page fits on the screen.

First Page — Displays the first page of the report.

Previous Page — Displays the previous page of the report.

Next Page — Displays the next page of the report.

Last Page — Displays the last page of the report.

Printer Setup — Displays the standard Windows Print Setup dialog box. From this dialog box, a target printer can be chosen and its settings can be altered before printing.

Print — Sends the report to the selected printer.

Save Report — Displays the **Save Report** dialog box allowing the report to be saved to a QuickReport (*.QRP), Comma Separated Text (*.CSV), HTML (*.HTM), or Excel spreadsheet (*.XLS) format.

Load Report Previously saved reports can be loaded into the Print Report Preview window. Only reports saved to the QuickReport (*.QRP) format can be reloaded.

Download

Selecting Download (using the **Download** button or the **Download Program** option in the Utility menu) sends the parameters displayed on the Test Parameters page to the specified memory location in the DV-III Ultra Rheometer. After a few seconds, a dialog box appears indicating that the rheometer received the parameters.

If no dialog box appears, see **Appendix I - Troubleshooting** - to help determine why the parameters were not received by the rheometer.

Run

Selecting **Run** and a program number (using the **Run** button or the **Run Program** option in the **Utility** menu) directs the DV-III Ultra Rheometer to run the selected program.

Note: The program numbers refer to the memory locations in the DV-III Ultra Rheometer.

After a few seconds, the DV-III Ultra tells EZ-Yield that the test has begun. Once all optional preparation steps have been performed by the rheometer, EZ-Yield begins to receive data as the test runs. If the On-Line Plotting option is selected in EZ-Yield, the Graph page is automatically displayed as soon as the first data point is received. Data are plotted as received, while being displayed in the Data/Results section of the Data page.

If the rheometer is running the test and no data appears, see **Appendix I - Troubleshooting** - to help determine why EZ-Yield is not receiving the data. When the test is complete, a dialog box is displayed asking you to save the freshly collected data and results.

Help

Selecting Help (using the Help button or the Help option on the menu) displays the on-line help system.

Exit

Selecting Exit (using the Exit button or the Exit option on the File menu) exits the EZ-Yield software. If test parameters or data have not yet been saved, a dialog box appears informing you of this fact and giving you the opportunity to save the pertinent information.

COM Port

Select the COM Port on the host PC (sometimes referred to as a serial port or an RS-232 port) to which the DV-III Ultra Rheometer is attached. COM1 through COM4 may be selected. Selecting NONE disconnects the rheometer from the host PC.

Note: Simply selecting the COM port is only part of what is needed for a valid connection between the rheometer and the host PC. Ensure that the appropriate cable is in use (Brookfield part # DVP-80) and that it is connected properly. See **Appendix H - Communications** - for more information regarding the DV-III Ultra to host PC connection.

IV.6.3 Data Page

Use this page to load, save, print, and export data taken with the DV-III Ultra Rheometer. The file name (including path) of the data currently loaded is displayed at the top of the page.

The test parameters used to collect the data are displayed on the left of the page in the box labeled “Parameters Used To Collect Data”.

Data is displayed on the right of the page in the box labeled “Data/Results”. All recorded data points are displayed in the table.

Time Elapsed time since the beginning of the run cycle is shown here in units of hours, minutes, seconds, and thousandths of seconds (H:MM:SS.SSS). Data is taken at an interval equal to the Base Increment parameter.

% Torque This field displays actual measured % Torque value

Delta % Torque This field displays the difference between the current % Torque value and the previous % Torque value. As the sample approaches its yield point, the Delta % Torque should begin to decrease. If the Torque reduction for the test is 100%, the yield point occurs when the % decrease in torque = 100.

Stress This field displays the calculated stress for each data point. The final Stress value (i.e. the Stress at the yield point) is the resultant yield stress. See Appendix D - Calculations - for the equation used to calculate stress.

Strain This field displays the Apparent Strain placed on the sample for each data point. The Apparent Strain is the angular distance that the spindle rotates in the sample. **See Appendix D2 - Spindle Codes and Speeds for Yield Tests** - for the equation used to calculate strain.

The following are displayed in the line immediately below the Data/Results table:

1. The number of the highlighted record
2. The total number of data records
3. The date and time that the highlighted record was taken.

The test results are displayed below that line.

Test Result There are five (5) possible test results:

1. Test Passed
2. Test Failed due to an Under-Range condition
3. Test Failed due to an Over-Range condition
4. Test Failed due to a User-Limit violation
5. Test Failed due to a user cancellation

See Section IV.6.6.3 - Test Results - for more information regarding these possible results.

Temperature The temperature recorded at the conclusion of the test is displayed here in °C.

% Torque @ Yield The %Torque value at the yield point is displayed here.

Yield Stress The stress at the yield point is displayed here in Pascals (Pa).

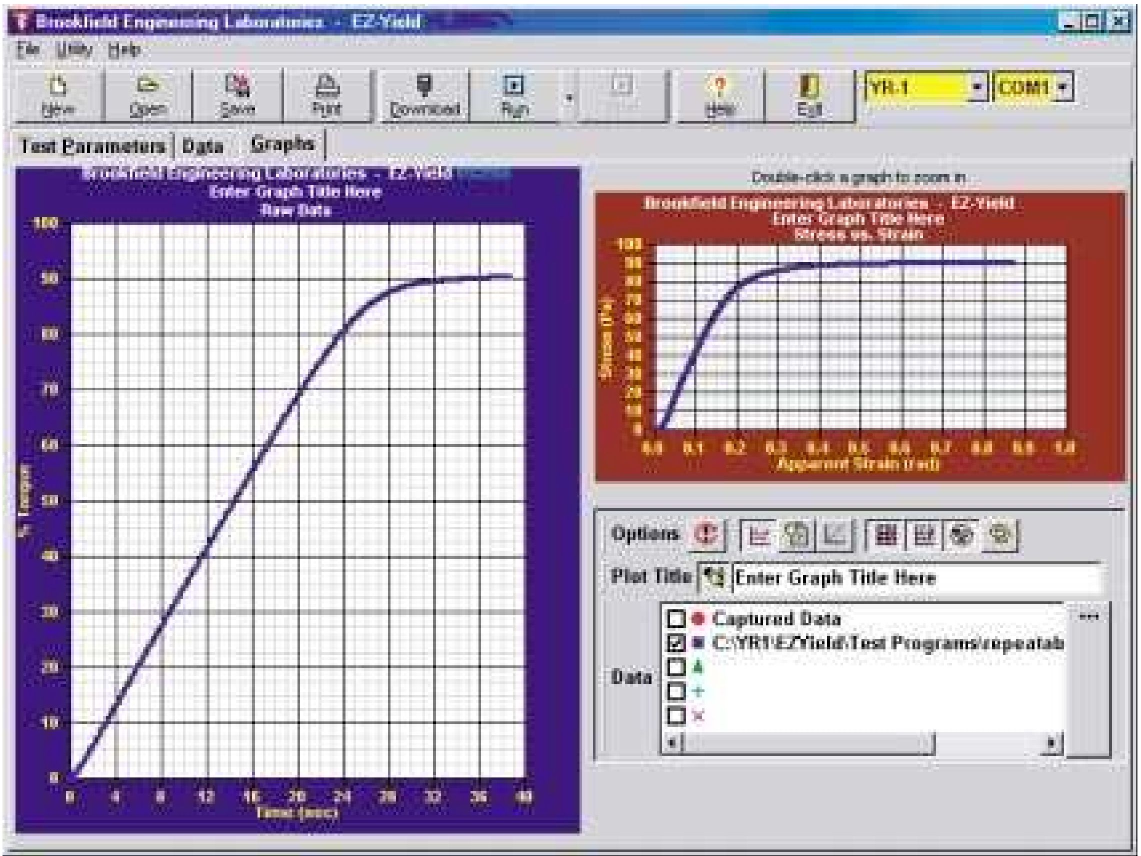
Clicking the **EXPORT** button displays the **Print Preview** window. From the **Print Preview** window, the **SAVE REPORT** button can be clicked to allow you to save the report to various other file formats.

Note: The **Save Report** dialog box allows reports to be saved to in QuickReport (*.QRP), Comma Separated Text (*.CSV), HTML (*.HTM), and Excel spreadsheet (*.XLS) formats.

See Section IV.6.6.2 - Printing a Program - for more information regarding this window.

All open, save, and print operations selected while this page is displayed will open, save, and print data, but NOT test parameters.

IV.6.4 Graphs Page



Use this page to graphically display yield test data both on-line (i.e. while the test is running) and after it is collected.

Two (2) graphs are displayed on this page.

1. <u>% Torque vs. Time</u>
   This graph displays the % Torque values versus the time (since the beginning of the Run cycle) at which the data points were collected. Torque is displayed in units of % of full scale range while the Time is displayed in seconds.
2. <u>Stress vs. Apparent Strain</u>
   This graph displays the calculated Stress values versus the calculated Apparent Strain values for each data point collected. Stress is displayed in units of Pascals (Pa) while Apparent strain is displayed in units of radians (rad).

See Appendix D2 - Spindle Codes and Speeds for Yield Tests - for more information regarding these calculated values.

Double-clicking on either graph enlarges or zooms that graph and displays it on the left side while shrinking the other graph and displaying it on the right.

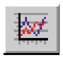
The three (3) toolbars on the right side of the page can be resized and moved within the confines of the toolbar box by clicking and dragging the toolbar sizer located to the left of the toolbar title.

Graphs Page Options Toolbar

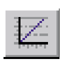
Note: Many of the Options buttons are two (2) state buttons. Two (2) state buttons have two (2) positions: depressed and un-pressed. A visually depressed button indicates the option is turned ON while a visually un-pressed button indicates that the option is OFF. Clicking a two (2) state button causes its state to change, thereby turning an option ON and OFF.

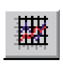
The following is a functional description of each of the buttons on the Options toolbar of the Graphs page:

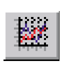
 **Replot** — Click this button to refresh both graphs. Any time another option is changed, the **REPLOT** must be clicked to see those changes.

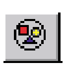
 **On-Line Plotting** — This is a two (2) state button. Turning this option ON causes data to be plotted on both graphs during the run cycle of a yield test. Keep in mind that the DV-III Ultra Rheometer must be communicating with the host PC and EZ-Yield™ in order for this data to be plotted.

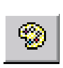
 **Show Data Values** — This is a two (2) state button. Turning this option ON causes a hint box to be displayed whenever the mouse cursor is placed on the Stress versus Apparent Strain graph within the data boundaries of the graph. This hint box displays the Time, % Torque, and Stress of the position pointed to by the cursor for the % Torque vs. Time graph. It displays Stress and Apparent Strain of the position pointed to by the cursor for the Stress vs. Strain graph.

 **Calibration Data** — This is a two (2) state button. Turning this option ON causes the straight line, representing the calibration information of the rheometer, to be displayed along with the user data on the % Torque vs. Time graph.

 **Major Grid Lines** — This is a two (2) state button. Turning this option ON displays the major grid lines (those with a numerical representation on each axis) to be displayed.

 **Minor Grid Lines** — This is a two (2) state button. Turning this option ON displays the minor grid lines (those without numerical representation on each axis) to be displayed.

 **Data Markers** — This is a two (2) state button. Turning this option ON displays a data marker (i.e. a colored shape matching that in the Data legend) for each data point.

 **Color Printouts** — This is a two (2) state button. Turning this option ON allows graphs to be printed in color if an appropriately equipped printer is connected and properly configured in Windows.

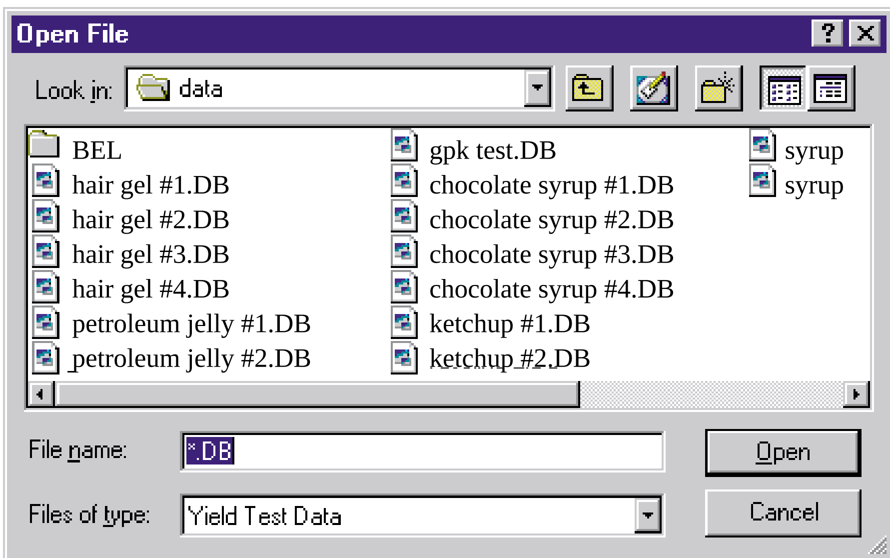
Use the **PLOT TITLE** two (2) state button to toggle between a user supplied graph title (the same title is used on both graphs) or a title indicating the yield test results. When the **PLOT TITLE** button is depressed, the user supplied title is used. You may delete the default "Enter Graph Title Here" message and type in a new one.

Note: The Replot button must be clicked after making any changes to the state of the **PLOT TITLE** button or to the user-supplied text.

Graphs Page Legend Toolbar

This toolbar allows you to select multiple data sets to plot (up to 5) and select which color and marker to use for each data set. To select a data set to be plotted, click the box to the left of the marker so that a check mark is displayed. To un-select a data set so that it is not plotted, click the box to the left of the marker so that a check mark is NOT displayed. Once the appropriate data sets to be plotted are selected, click the **REPLOT** button in the **Options** toolbar to update the graphs.

The top line in the toolbar always displays **Captured Data** and cannot be changed. Select this data set to graph freshly collected (i.e., unsaved) data. The bottom four lines are used to select and load data from disk files. To select a file either double-click the intended line or select the line with a single click, then click the **BROWSE** button (i.e. the button with the three (3) dots or ellipsis). The **Open File** dialog box appears allowing you to navigate to and select the desired data file to graph.



Note: The **REPLOT** button must be clicked after making any changes to the state of the **Data Legend** toolbar in order for the changes to be seen on the graphs.

On the graphs, the data is plotted using the color specified for the selected data sets in the **Data Legend** toolbar. In addition, if the **Data Markers** option is turned ON, the appropriate data marker from the legend is displayed on each graph for each data point.

IV.6.5 Examples of Yield Test Programs

IV.6.5.1 Example Application: Hair Gel

The test method and data shown in Figure 61 show an analysis of yield from a typical hair gel product. It is usually desirable to measure yield on products without transferring them from their containers because doing so would "pre-shear" the material. Hair gel is normally sold in shallow jars, as was the case with this sample. The V-73 vane spindle was selected because it is the smallest and could be completely immersed in the shallow jar. An RVDV-III Ultra Rheometer was used for the test.

The spindle was immersed to the primary immersion mark and pre-shear was turned off. Zero was turned on and a nominal "zero" speed of 0.1 rpm was chosen. This allows the motor to reset the torque to zero prior to running the test. Occasionally, the spindle rotates a bit while being lowered into the sample. This moves the torque sensor away from zero. It is usually desirable to reset it to zero before starting the measurement. Run speed was set to 0.1 rpm. Often, 0.5 rpm is selected and is a good starting point when establishing test parameters. In this case, a slow test was desired so test speed was set intentionally low. Low and high yield limits (for Q.C.) were ignored.

This test resulted in a good mid-range torque of 44.5% at the yield stress, which was reported as 44.38 Pa.

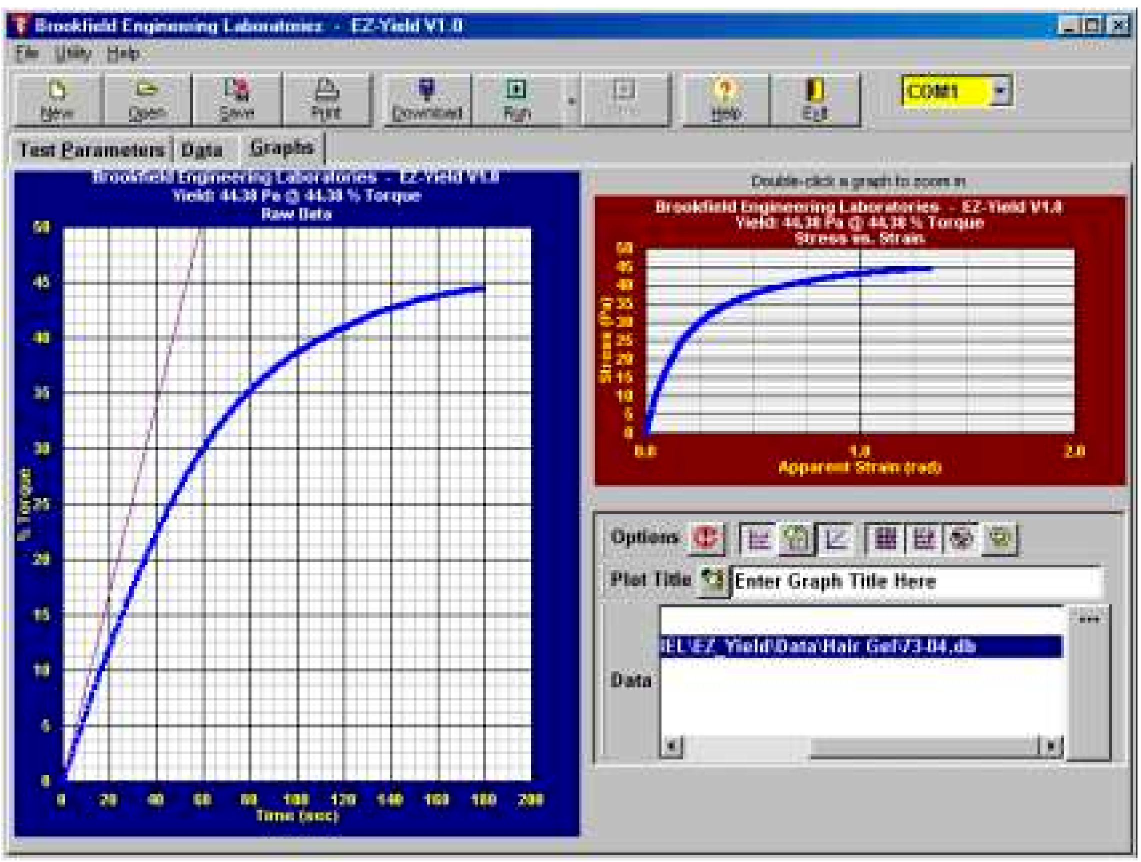


Figure 61: Hair Gel Sample

IV.6.5.2 Example Application: Hand Cream

Hand cream is typically packaged in a small jar. The small V-73 spindle did not provide enough torque signal (less than 10% on the RVDV-III Ultra Rheometer) so the larger V-72 spindle was used. The jar was too shallow to completely immerse the spindle so the secondary immersion mark was used. This provided just enough torque at the yield point for a satisfactory test. Pre-shear was turned off. Zero was turned on and a low test speed of 0.1 rpm was selected. Torque reduction was left at the default setting of 100%. Q.C. limits were turned off (by selecting zero for their values). The base increment (data interval) was left at the default 1,000.

Upon conclusion of the test, individual data points were widely spaced because yield was reached so quickly. In this case, either a lower *Run Speed* might be desired or a smaller *Base Increment*. Both would increase the density of data points and result in a more accurate or repeatable test result.

The yield stress resulting from this test was reported as 190.32 Pa.

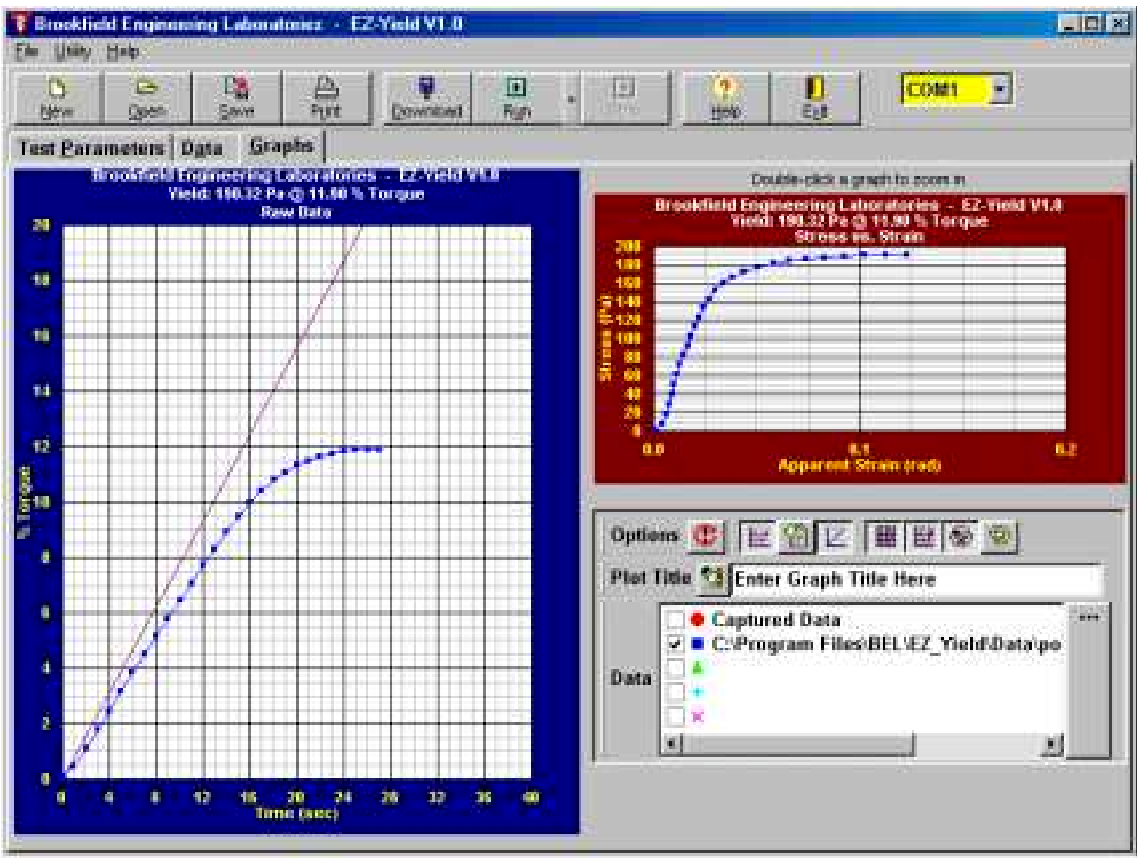
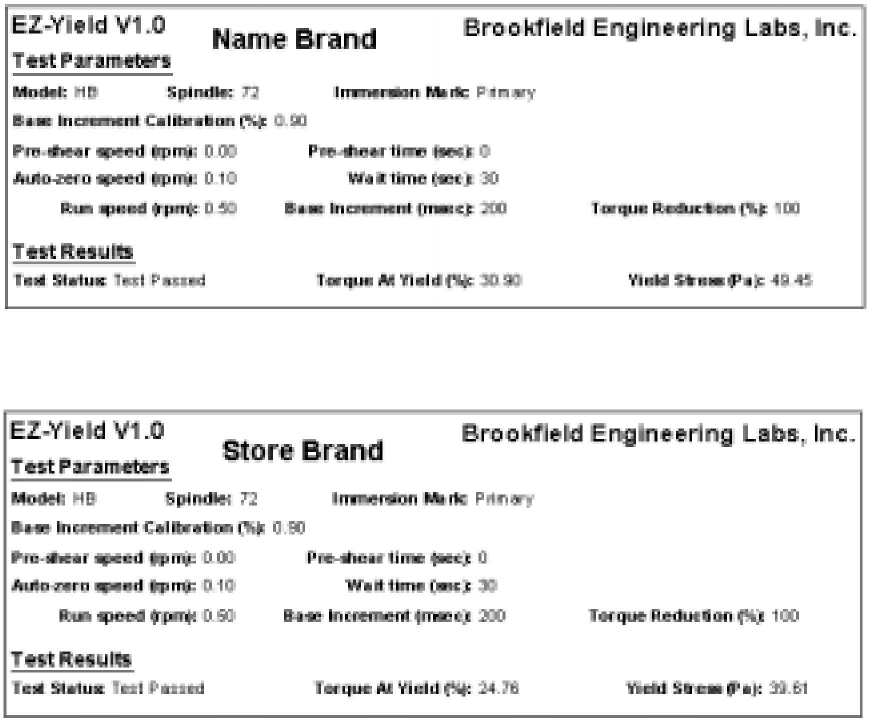


Figure 62: Hand Cream Sample

IV.6.5.3 Example Application: Ketchup

In this example, a name brand ketchup was tested and compared to a store brand ketchup. Visual examination of both products could distinguish no noticeable differences. Both tests were run under exactly the same conditions using an HBDV-III Ultra with spindle V-72. See Figure 63 for the test set up.

Significant differences between the name brand and the store brand can be characterized by such a test as shown in Figure 64. Both the slope of the stress/strain curves and the yield points are clearly different.


EZ-Yield V1.0 **Name Brand** Brookfield Engineering Labs, Inc.

Test Parameters

Model: HB **Spindle:** 72 **Immersion Mark:** Primary

Base Increment Calibration (%): 0.90

Pre-shear speed (rpm): 0.00 **Pre-shear time (sec):** 0

Auto-zero speed (rpm): 0.10 **Wait time (sec):** 30

Run speed (rpm): 0.50 **Base Increment (msec):** 200 **Torque Reduction (%):** 100

Test Results

Test Status: Test Passed **Torque At Yield (%):** 30.90 **Yield Stress (Pa):** 49.45

EZ-Yield V1.0 **Store Brand** Brookfield Engineering Labs, Inc.

Test Parameters

Model: HB **Spindle:** 72 **Immersion Mark:** Primary

Base Increment Calibration (%): 0.90

Pre-shear speed (rpm): 0.00 **Pre-shear time (sec):** 0

Auto-zero speed (rpm): 0.10 **Wait time (sec):** 30

Run speed (rpm): 0.50 **Base Increment (msec):** 200 **Torque Reduction (%):** 100

Test Results

Test Status: Test Passed **Torque At Yield (%):** 24.76 **Yield Stress (Pa):** 39.61

Figure 63

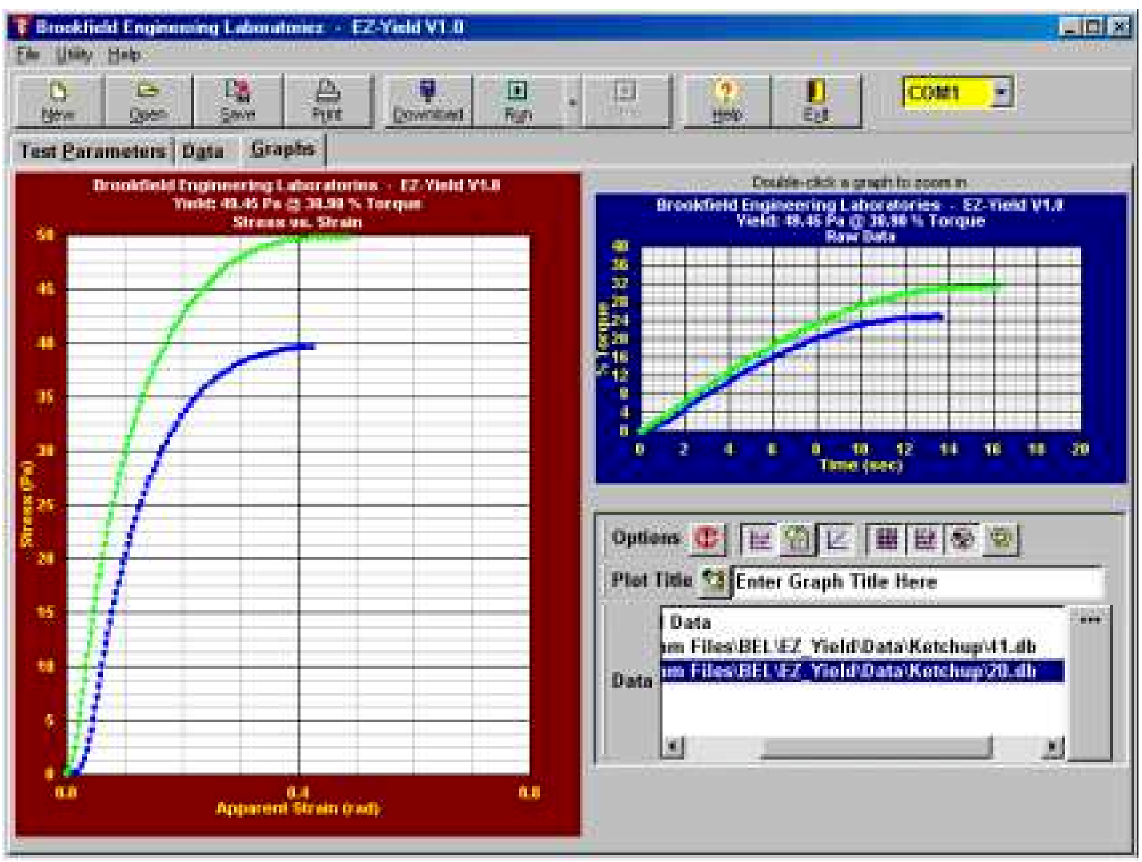


Figure 64: Ketchup Comparison Samples

IV.6.5.4 Torque vs. Time for Various Food Products

Figure 65 shows graphs of Torque (%) vs. Time (seconds) and Stress (Pa) vs. Strain for several different materials: cream cheese, mayonnaise, pudding and ketchup. Experimentally determining an appropriate combination of torque range, spindle and speed for testing each material ensures that the torque readings are within the recommended 10-100% on the torque scale. If a sample has a relatively low torque reading for the yield (<10%), then try using a faster speed or larger spindle to get a larger torque reading. Alternatively, a DV-III Ultra Rheometer with a more sensitive spring could be used as well; e.g., RVDV-III Ultra instead of HBDV-III Ultra.

You will need different spring torques in order to test a wide range of products that have vastly different yield characteristics. The spring torque of the HB is eight times greater than that of the RV and the 5xHB torque is 40 times greater than that of the RV (or five times that of the HB).

An advantage of the EZ-Yield™ software is that multiple samples can be compared graphically on the same set of axes for easy and rapid evaluation of yield behavior.

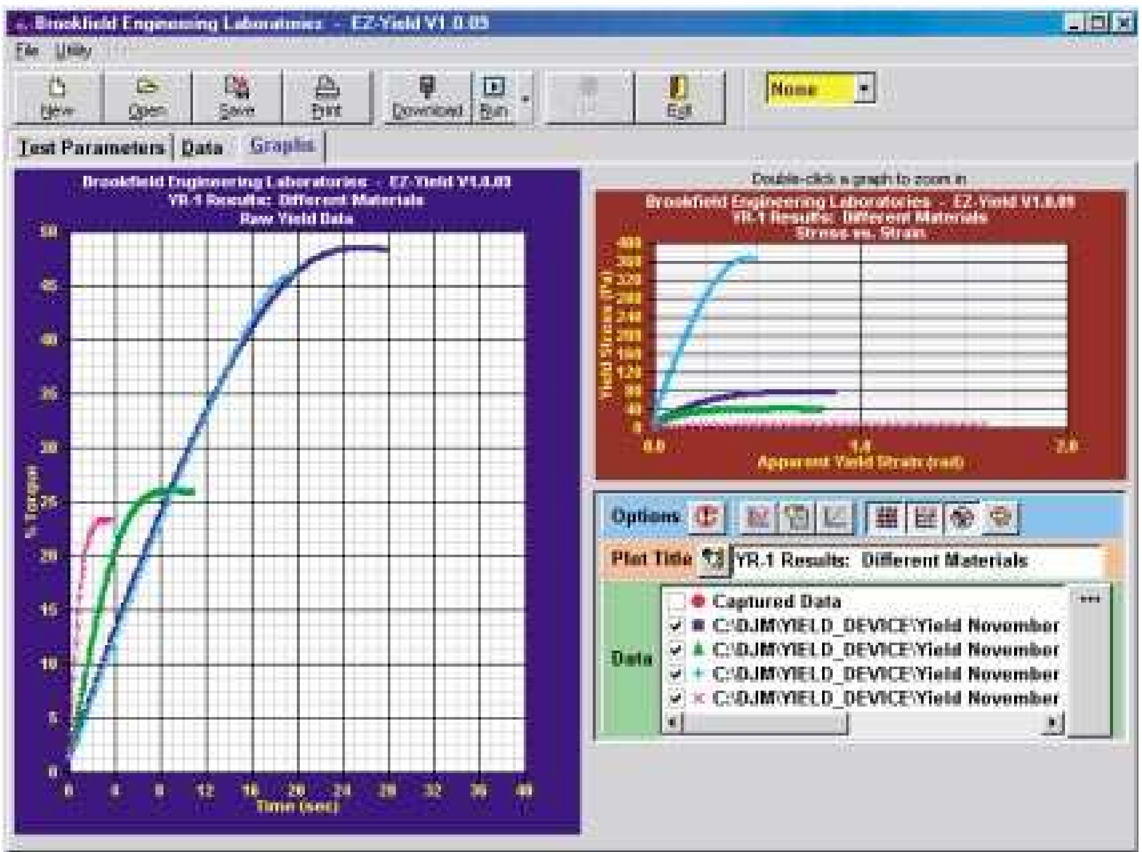


Figure 65: Torque vs. Time Graphs for Various Food Products

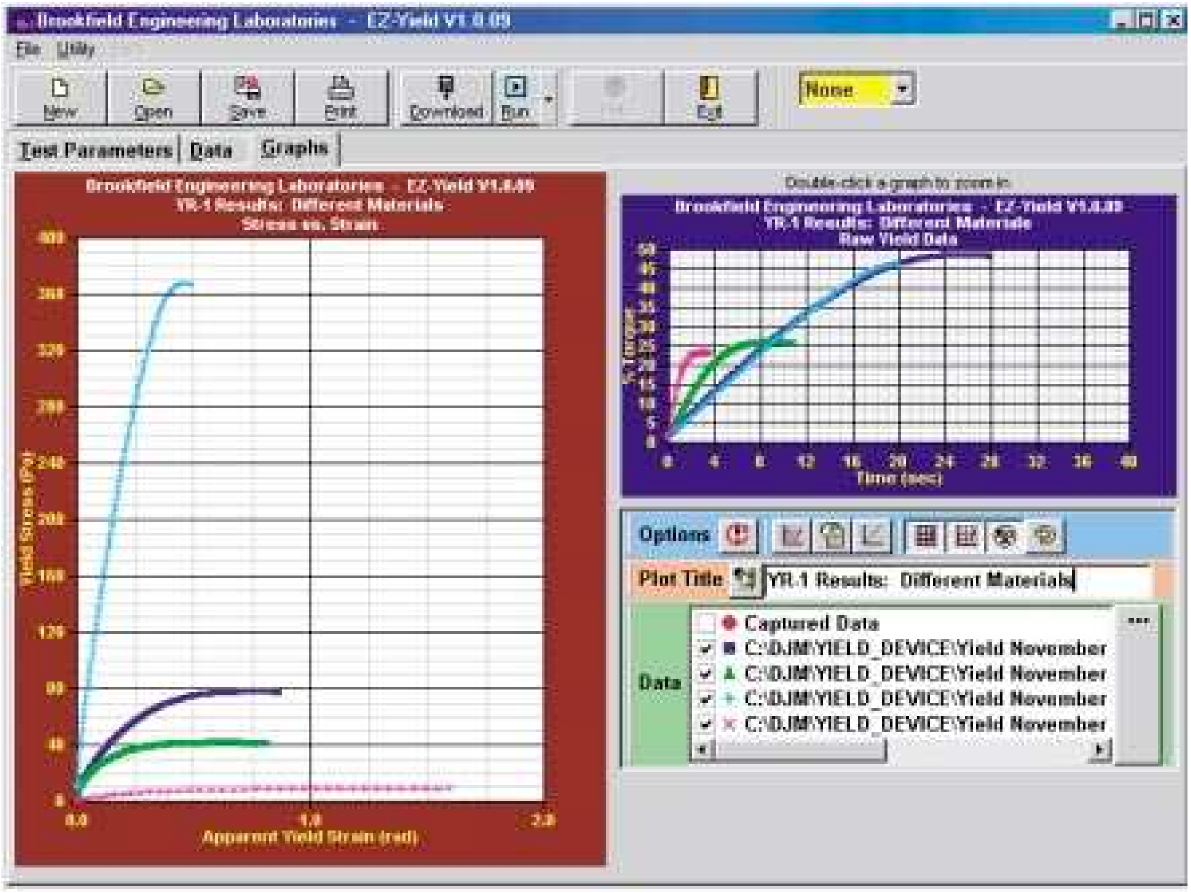


Figure 66: Stress vs. Strain Graphs for Various Food Products

IV.6.6 Running Yield Test Programs in Stand-Alone Mode

This section explains how to use the DV-III Ultra Rheometer in standalone mode. There are no test programs stored in instrument memory when the DV-III Ultra is shipped from Brookfield. Therefore, you will need to review the preceding section on the EZ-Yield™ Software which explains how to create test programs. Once a test program has been downloaded from your PC to the DV-III Ultra, you will be able to run tests by following the instructions in this section.

♦ Yield Programs Menu

Yield test operations are accessed by pressing the "4" key when in the **PROGRAM MODES** menu. The following screen is immediately displayed:

```
    YIELD TEST
#5: Sample Tst
ENTER = Run Test
PRINT = Print Test
```

Figure 67

The program number and the program name (in this case, “#5: Sample Tst”) are displayed on the second line. The program name flashes to indicate that it is the currently chosen test.

Pressing the **OPTIONS/TAB** key scrolls through the program numbers (0-9) and displays the corresponding program name.

Note: If the program number chosen has no program loaded, the word EMPTY is displayed as the program name.

Pressing the **MOTOR ON/OFF/ESCAPE** key from this screen returns the display to the **PROGRAMS MODE** menu.

Running Yield Programs

When the **ENTER** key is pressed while a valid program is chosen in the Yield Programs Menu (Figure 67), the following screen is displayed:

```
#5: Sample Tst

   USE SPINDLE #71
    PRESS ENTER
```

Figure 68

The spindle number for the current yield program is displayed on line 3. Prepare the sample, attach the appropriate spindle and lower it into the sample before continuing. Be sure to use the correct immersion mark.

Pressing the **ENTER** key with Figure 68 displayed performs the following:

- The test is started.
- The pre-test information (test parameters) is sent to both the serial (RS-232) port and to an attached printer via the parallel printer port. See section IV.6.6.1 for a description of the pre-test information sent to the printer.
- The program number being run is recorded so that it will be the first program shown the next time the Yield Programs Menu (Figure 67) is displayed.

Pressing the **MOTOR ON/OFF/ESC** key at this screen causes Figure 67 to be displayed.

After ensuring the appropriate spindle is attached and pressing the **ENTER** key, the various Run screens are displayed. The top line of the Run screens always displays the program and spindle number as follows:

```
#5: Sample Tst  S:71

--------------------
--------------------
```

Figure 69

where the program and spindle numbers in use are displayed on the top line. The bottom lines of the Run screens vary depending upon the options selected in the program.

Figure 70 is displayed when a Pre-Shear speed and time were selected. If the current program requires a Pre-shear step, the spindle will rotate at the selected speed for the selected time. The field after the word “RPM” is the speed of the pre-shear (in rpm) while the field after the word "Time" is a time (in seconds) that counts down from the user defined pre-shear time. A Pre-shear step allows the sample to be conditioned before the actual test is started. In Figure 70, the pre-shear speed is 10 rpm and the pre-shear time is 30 seconds.

```
#5: Sample Tst  S:71

PRE-SHEARING...
RPM: 010  TIME: 0030
```

Figure 70

Figure 71 is displayed when a Zero operation was selected. If the current program requires a Zero step, the spindle rotates backwards until the % torque reading is zero (0). The field after the word “RPM” is the speed (in rpm) while the field after the word "Torque" is the current % torque reading. A Zero step ensures that each test is started from 0% torque. In Figure 71, the speed is 0.2 rpm and the torque reading is momentarily at 2.4% as it decreases back to 0%.

```
#5: Sample Tst  S:71

ZEROING...
RPM: 0.2  TORQUE: 2.4
```

Figure 71

Figure 72 is displayed when a **Wait Time** was selected. A **Wait Time** step allows for a user-defined time delay before starting the test. The field after the word “Time” is the time, in seconds, that counts down from the user-defined wait time.

```
#5: Sample Tst  S:71

WAITING...
TIME: 0010
```

Figure 72

Figure 73 is always displayed during the actual test, after the aforementioned preparation steps have been completed. During this portion of the test, data is continuously sent out the serial (RS-232 port) located on the rear panel of the instrument. This data is for use with the accompanying EZ-Yield™ software. See Appendix H for more information regarding this data.

```
#5: Sample Tst  S:71

   RUNNING YIELD
```

Figure 73

If the **ESC** key is pressed at any time during the yield test run, the following is displayed:

```
#5: Sample Tst  S:71

USER TERMINATED TEST
 PRESS ESC OR ENTER
```

Figure 74

At this point, pressing the **ENTER** key starts the test again starting from whichever **Run** screen is applicable (Figures 70 through 73). Pressing the **ESC** key returns the display to that of Figure 68, reminding you which spindle should be used.

When the test is allowed to run to completion, one of the following screens (Figures 75 through 78) are displayed:

Figure 75 is displayed when the yield test passes. The % torque and stress (in Pa) at the point of yield are displayed along with the temperature (in °C) at the conclusion of test.

```
#5: Sample Tst  S:71
    TEST COMPLETE
%=73.56    TEMP=025.5
YIELD=0726.4 Pa
```

Figure 75

Figure 76 is displayed when the yield test fails due to an over-range error.

```
#5: Sample Tst  S:71
    YIELD ERROR
     OVER-RANGE
 PRESS ESC OR ENTER
```

Figure 76

Figure 77 is displayed when the yield test fails due to an under-range error.

```
#5: Sample Tst  S:71
    YIELD ERROR
    UNDER-RANGE
 PRESS ESC OR ENTER
```

Figure 77

Figure 78 is displayed when the yield test fails due to the resultant yield stress exceeding the user defined limits. The actual yield stress measured during the test is displayed in the first field to the right of the "Yield=", while the limiting value causing the failed test (high or low yield stress) is displayed in the second field.

```
#5: Sample Tst  S:71
    LIMIT ERROR
YIELD=726.436 (200.0)
 PRESS ESC OR ENTER
```

Figure 78

After screen 75 through 78 is displayed, pressing the **MOTOR ON/OFF/ESC** key returns the screen to Figure 68. At this point, after ensuring the appropriate spindle is attached, the **ENTER** key can be pressed to run the test again. Pressing the **ENTER** key when any of these screens is displayed immediately runs the test again and displays the first appropriate Run screen (Figures 70 through 73). When the test is complete, the results are sent out of both the parallel and serial (RS-232) ports.

IV.6.6.1 Data Output

Pre-Test Output

When a test is begun, the following information is sent out both the parallel and serial (RS-232) ports in the format shown with each line terminated by a CR/LF (i.e., a Carriage Return/Line Feed command):

Date	: ....
Sample Information	: ....
Operator	: ....

Model	:	RV			
YD1:Spindle	:	71/001	YD2:Pre-shear (rpm/secs)	:	0.1/0010
YD3:Zero (rpm)	:	0.05	YD4:Wait Time (secs)	:	0010
YD5:Run Speed (rpm)	:	0.4	YD6:Base Inc. (msec)	:	0200
YD7:Torque Red. (%)	:	020	YD8:Under-Range Red. (%)	:	050
YD9:Average (readings)	:	05	YDA:Limits (Low/High)(Pa)	:	00200/01000
Base Inc. Cal. (%)	:	1.55	Temperature (°C)	:	25.5
Test Name	:	Sample Test	Slot Number	:	01

Notes: 1) The Base Inc. Cal. (Base Increment Calibration) number is the change in torque expected for the Base Inc. (Base Increment) selected using the calibration data stored in the instrument.

2) The YDx values are for use with the EZ-Yield™ software and can be ignored.

Post-Test Output

At the conclusion of the test, one of the following six (6) messages is sent to both the parallel and serial (RS-232) ports:

1. Test (test number: test name) Complete
   Yield Stress (Pa) = 196.53 % Torque @ Yield = 78.6 Temperature = 25.5C
   Test Passed
2. Test (test number: test name) Complete
   Test Failed = Under-range (displayed if test fails due to an under-range condition; see Section IV.6.6.3)
3. Test (test number: test name) Complete
   Test Failed = Over-range (displayed if test fails due to an over-range condition; see Section IV.6.6.3)
4. Test (test number: test name) Complete
   Yield Stress (Pa) = 196.53 % Torque @ Yield = 78.6 Temperature = 25.5C
   Test Failed = Yield Stress Below Low Limit (displayed if test fails due to resultant yield stress being below low limit; see Section IV.6.6.3)
5. Test (test number: test name) Complete
   Yield Stress (Pa) = 196.53 % Torque @ Yield = 78.6 Temperature = 25.5C
   Test Failed = Yield Stress Above High Limit (displayed if test fails due to resultant yield stress being above high limit; see Section IV.6.6.3)
6. Test (test number: test name) Cancelled
   Test Failed = Cancelled By User (displayed if test fails due to cancellation by the user)

IV.6.6.2 Printing A Program

Pressing the **PRINT** key with a valid program selected in the Yield Programs Menu (Figure 67) displays the following screen:

```
#5: Sample Tst

READY PRINTER THEN
PRESS PRINT KEY
```

Figure 79

Pressing the **ENTER** key at this screen causes the selected program information to be sent out the parallel printer port (located on the rear panel of the instrument).

Pressing the **MOTOR ON/OFF/ESC** key at this screen returns the display to that of Figure 67.

If, after pressing the **PRINT** key with Figure 79 displayed, a printer error is detected (Ex: printer being off-line), the following screen is displayed:

```
#5: Sample Tst
PRINTER IS NOT READY
ENSURE PRINTER IS
ONLINE-PRESS ANY KEY
```

Figure 80

Pressing any key at this point returns the display to that of Figure 79.

Pressing the **ENTER** key also records the program number being printed so that it will be the first program shown the next time PRINT TEST is selected from the default screen (Figure 67).

After printing is complete, the screen returns to the display shown in Figure 67.

Note: Printing can only be accomplished using the parallel printer port. The serial port (RS-232) is strictly used for downloading programs and/or collecting yield data using the accompanying EZ-Yield™ software.

Test programs are printed in the following format:

```
                    Program Number/Name: #5/Sample Tst

Operator:                                                Date:

Spindle Number: 71                    Multiplier  :  002
---------------------------------------------------------
Preparation Parameters
----------------------

Pre-Shear Speed (rpm):  10           Pre-Shear Time (sec): 0030

Zero Speed (rpm): 0.2                Wait Time (sec): 0010

Run Parameters
--------------

Run Speed (rpm): 0.5                 Base Increment (msec): 0100

Torque Reduction (%): 100

Average (readings): 05               Limits (Low/High) (Pa):0090/0100
```

IV.6.6.3 Test Results

Test Passed

When a yield test runs successfully, the resultant Yield Stress (Pa), the % Torque value at the time of the yield and the Temperature at yield are printed.

```
Test (#5: Sample Tst) Complete
Yield Stress (Pa) = 726.36 % Torque @ Yield = 73.56   Temperature = 25.5C
```

In addition, the results are shown on the display as follows:

```
#5: Sample Tst  S:71
    TEST COMPLETE
%=73.56    TEMP=025.5
YIELD=0726.4 Pa
```

Figure 81

Test Failed

Some of the reasons a yield test may fail are as follows.

Under-Range Condition

Only yield measurements in the % Torque range from 10% to 100% will be accepted. If a Yield

Stress is reached before the % Torque value is 10%, an Under-Range error causes the test to end.

The following is sent to an attached printer:

```
Test (#5: Sample Tst) Complete
Test Failed = Under-range
```

The following screen is displayed:

```
#5: Sample Tst  S:71
    YIELD ERROR
    UNDER-RANGE
PRESS ESC OR ENTER
```

Figure 82

Under-Range Error

The yield stress (Torque Reduction Value) is reached before the absolute % torque reaches 10%. When a test is started, once the absolute torque is greater than zero (0), the second, third, and fourth Delta Torques (also known as the Base Increment Torque) are averaged. The first three (3) Delta Torques after 9% absolute torque is reached are also averaged. An under-range error occurs if the second average falls below the first average by 15% or more.

Over-Range Condition

Only yield measurements in the % Torque range from 10% to 100% will be accepted. If Yield Stress is not reached before the measured % Torque value reaches 100%, an Over-Range error causes the test to end.

The following is sent to an attached printer:

```
Test (#5: Sample Tst) Complete
Test Failed = Over-range
```

The following screen is displayed:

```
#5: Sample Tst  S:71
    YIELD ERROR
    OVER-RANGE
PRESS ESC OR ENTER
```

Figure 83

User-Defined Limit Condition

When an upper and lower limit has been imposed upon the Yield Stress by the user, any resulting Yield Stress that falls outside these limits causes the test to fail with a Limit Violation Condition. One of the following messages is sent to the attached printer dependent upon which limit was violated:

Low Limit Violation

```
Test (#5: Sample Tst) Complete
Yield Stress (Pa) = 196.53 % Torque @ Yield = 27.4    Temperature = 250.5C
Test Failed = Yield Stress Below Low Limit
```

High Limit Violation

```
Test (#5: Sample Tst) Complete
Yield Stress (Pa) = 996.53 % Torque @ Yield = 78.6    Temperature = 250.5C
Test Failed = Yield Stress Above High Limit
```

The following screen, showing first the actual yield stress followed by the violated limit value, is displayed:

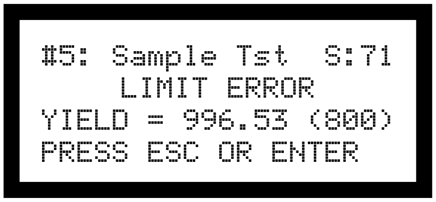


Figure 84

Test Cancelled By User

When a test is cancelled by the user, either by pressing the ESC key during a running test or by canceling the test via the EZ-Yield™ software (See Section IV.6.2), the following is sent to the attached printer:

```
Test (#test number: test name) Cancelled
Test Failed = Cancelled By User
```

The following screen is displayed:

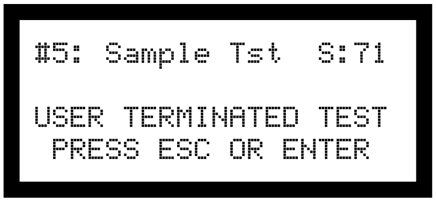


Figure 85

V. OPTIONS

The DV-III+ Options Menu allows the user to execute temperature control commands and special time tests. General rheometer settings are also accessed from this menu. The Options Menu is shown in Figure 86. Selections are made by pressing the appropriate number key.

```
   OPTIONS MENU
1 SETUP   4 SET TEMP
2 PRINT   5 DATA
3 ALARMS  6 TIMED
```

Figure 86

V.1 Set Up

1. Change the units of temperature. The change is selected by pressing the1 key. The change must be confirmed by pressing the **ENTER** key.

2. Change the units of viscosity and shear stress (CGS System: cP, D/cm^2) (SI System: mPa•s, N/m^2).

   The change is selected by pressing the 2 key. The change must be confirmed by pressing the **ENTER** key.

3. Change communication status with external Brookfield temperature controller. An "off" indication means that there is no communication with a controller. Selecting this option will make the DV-III Ultra try to establish communication. When communication is established, "off" will be change to "on". Temperature control will always be set to "off" when the DV-III Ultra is turned on.

   When communication is established, Line 1 of the default screen will be modified. The temperature field will show "CTLR" in place of "TEMP".

   Communication may only be established with Brookfield controllers:

Thermosel Controllers:	HT-106, HT-104
Bath Controllers:	HT-107, HT-105

   The change is selected by pressing the 3 key. Subsequent key presses required will be indicated on the display.

4. Change the temperature display by offsetting the measured temperature to agree with an external temperature measurement device. The adjustment will be indicated by flashing temperature units (F or C) on the default screen (**Figure 7**). The adjustment will be reset to 0.0 when the DV-III+ is turned on.

   The adjustment is entered using the number keys. The sign (+ or -) is selected using the **OPTION/TAB** key. The change must be confirmed by pressing the **ENTER** key.

V.2 Print

1. Change the time interval that is used when the DV-III Ultra is printing continuously. Data is entered in the format of MM:SS. For example: an interval of one minute and 30 seconds is entered as: 01:30.

   The change is initiated by pressing the 1 key. Once the time interval is input, it must be accepted by pressing the **ENTER** key.

2. Change the port to be used for printing, parallel or serial.

   The change is selected by first pressing the 2 key and then the appropriate key for the printing method. The change will be indicated by the position of the arrow on the right side of the display. The change is accepted by pressing the **ENTER** key.

   When selecting serial printing, the DV-III Ultra will also ask for handshaking status. Please see the instruction manual of the printer for specification.

   If the DV-III Ultra is communicating with an external temperature controller, serial printing will not be available(the controller uses the serial port).

3. Enter the date and time to be shown on printed data tables. Data is entered in the format of DD/MM/YY for date and HH:MM for time (24-hour clock). For example: 2:30 pm on January 15, 1999 is entered as 15/01/99, 14:30.

   The change is initiated by pressing the 3 key. Once the data is input, it must be accepted by pressing the **ENTER** key.

V.3 Alarms

There are three adjustable alarm settings: ***LO ALARM %***, ***HI ALARM %*** and ***MOTOR OFF %***. The values are set in the Set Alarms mode. Alarms are used to signal the operator that the fluid is out of the input specification. The alarms are set in % torque values, not Viscosity, Shear Stress or Shear Rate values. The range of values which may be entered for each alarm and their default values are:

LO ALARM		HI ALARM		MOTOR OFF	
Minimum value:	10%	Minimum value:	0%	Minimum value:	0%
Maximum value:	99.9%	Maximum value:	100%	Maximum value:	115%
Default value:	10%	Default value:	99.9%	Default value:	110%

The procedure for entering and enabling alarm values is as follows starting from the main screen:

1) Press the **OPTION/TAB** key to display the Options Menu.
2) Press the **NUMBER 3** key to display the Alarms Options screen.
3) Press the **NUMBER 1** key to View/Set Alarms.
4) Enter the LO ALARM % torque value. The new entry will overwrite the default values.
5) Press the **OPTION/TAB** key to move the cursor to the HI ALARM % field.

6) Enter the HI ALARM % torque value. The new entry will overwrite the default values.

7) Press the **OPTION/TAB** key to move the cursor to the MOTOR OFF % field.

8) Enter the MOTOR OFF % torque value. The new entry will overwrite the default value.

9) Press the **ENTER** key to accept the ALARM values.

10) Press the **NUMBER 3** key to enable (turn on) / disable (turn off) the alarms.

11) Press **ENTER** to accept the ALARM condition.

> **Note:** The LO ALARM is tripped after the % torque reading falls below the setting. The beeping may be shut off by either the % torque reading rising above the alarm setting or by pressing the ESCAPE key.
>
> The HI ALARM is tripped after the % torque reading goes above the alarm setting. The beeping may be shut off by either the % torque reading falling below the larm setting or by pressing the **ESCAPE** key.
>
> The MOTOR OFF is tripped after the % torque reading goes above the motor off setting. The DV-III Ultra stops rotating and the instrument beeps. Pressing any key turns off the beep.

V.4 Set Temperature

The DV-III Ultra can issue temperature control commands when an external temperature control device has been connected (see Section V.1). Selecting item #4 Set Temp in the Options menu will display the current setpoint. Enter the new setpoint by using the number keys and accept with the **ENTER** key. The temperature controller will begin using the new setpoint immediately upon the press of the **ENTER** key.

> **Note:** The sign of temperature is changed by using the arrow keys when the cursor is under the ± character. This is possible only with temperature bath controllers.

V.5 Data

The Review Data menu allows data review of the most recently completed speed set program. Data may be reviewed on the DV-III Ultra screen, on a serial printer, or both. The procedure for reviewing data after a test is as follows:

1) Run a DV-III or B.E.V.I.S. program.

2) Press the **OPTION/TAB** key.

3) Press the **NUMBER 5** key to display the Review Data screen.

```
   REVIEW DATA
1 = SCREEN ONLY
2 = SCREEN & PRINT
3 = PRINTER ONLY
```

Figure 87

4) Select one of the review modes: press "NUMBER 1" for DV-III Ultra screen only, "NUMBER 2" for DV-III Ultra screen and printer, or "NUMBER 3" for printer only. Note that you should select options 2 and 3 only if the printer is connected and "on-line."

5) If **NUMBER 1** or **NUMBER 2** were pressed, data from the first step is either displayed on the screen or displayed and printed. Press the **ENTER** key to scroll through and display/print the remaining step data.

```
cP=10800     STEP01
%=50.2   TIME=00:00
SS=0.0    TEMP=70.2
SR=0.0   RPM=100.0
```

Figure 88

6) If the **NUMBER 3** key (printer only) is selected, the DV-III Ultra prompts to "***READY PRINTER PRESS ANY KEY***" or "***PRINTER IS NOT READY TURN ON/PRESS ONLINE***". A sample of printed output is shown in Figure 89.

```
Sample Name:____________________________________________________

Operater Name:________________________

Date: 06/21/91  Time: 10:06  Model: 2R  Spindle: 31

#01 RPM=30.0  %=18.2  cP=970    D/CM2=100   1/SEC=10.2   T=22.1C  TIME=00:05
#02 RPM=40.0  %=24.3  cP=972    D/CM2=132   1/SEC=13.6   T=22.1C  TIME=00:05
#03 RPM=50.0  %=30.3  cP=969    D/CM2=164   1/SEC=17.0   T=22.1C  TIME=00:05
#04 RPM=60.0  %=36.3  cP=967    D/CM2=197   1/SEC=20.4   T=22.1C  TIME=00:05
#05 RPM=70.0  %=42.3  cP=966    D/CM2=230   1/SEC=23.8   T=22.1C  TIME=00:05
```

Figure 89

Review data notes:

1) The DV-III Ultra stores data from the most recent speed set program test. When a subsequent speed set program is run, any test data in memory will be overwritten and the previous data will be lost.

2) If the "Printer Only" option is selected, the date, time of day, model and spindle number are printed with the test results. The DV-III Ultra does not store time and date when it is

turned off; therefore, the time and date must be entered when printing the first time after start-up. See Section **V.2**.

3) The DV-III Ultra may be set-up to "*handshake*" (using **XOn/XOff** protocol) or not handshake when connected to a serial printer. If you opt to use the handshake mode, your printer is "*on line*", all cable connections are correct and your printer is set for handshake mode, then data should appear on your printer immediately. If it does not appear, and instead you see the message: "***PRINTER IS NOT READY TURN ON/PRESS ONLINE***" then you should turn the handshake option off. See Section **V.2**.

V.6 TIMED DATA COLLECTION

The DV-III Ultra offers three methods of time control that may be used independently of the control programs for making viscosity measurements described in IV.2 and IV.3. These techniques result in a single data point collected at the end of the test. If multiple points are required, the rheometer should be connected to a printer and set up with an appropriate print interval.

♦ TIME TO TORQUE

The DV-III Ultra will record the amount of time required to reach the specified % torque value at a single speed. Torque and speed are input using the number keys and **OPTIONS/TAB** key. The test will begin immediately upon the press of the **ENTER** key.

The test will end when the specified torque level is reached (in either an upward or downward direction). The data will be displayed as shown in Figure 90. The **SELECT DISPLAY** key can be used to view all measurement parameters. The **PRINT** key can be used to send a single data line to the printer.

```
RPM:OFF   SPINDLE:31
TEMP:72.1 F     PRTN
TIME TO TORQUE DONE
TORQ=89.4 % m=03S=22
```

Figure 90

Note: If continuous printing was used during the test, it will be suspended when viewing the test data or entering time to torque parameters.

♦ TIMED STOP

The DV-III Ultra will operate at a single RPM for a specified period of time. Time and speed are entered using the number keys and **OPTIONS/TAB** key. The test will begin immediately upon the press of the **ENTER** key.

The test will end when the specified time interval has elapsed. The data will be shown as displayed in Figure 91. The **SELECT DISPLAY** key can be used to view all measurement parameters. The **PRINT** key can be used to send a single dataline to the printer.

```
RPM:OFF   SPINDLE:31
TEMP:72.1 F     PRTN
TIMED STOP TEST DONE
TORQ=82.4 % m=02s=30
```

Figure 91

> **Note:** Continuous print mode may be used in conjunction with Timed Stop to print data throughout the time period. Printing will occur only during the test.

TIMED AVERAGE

The DV-III Ultra will collect a specified number of data points over a period of time and present the average reading (arithmetic average). Time, speed and the number of data points are entered using the number keys and **OPTIONS/TAB** key. The test will begin immediately upon the press of the **ENTER** key. **Non-averaged data will be displayed during the test**.

The test will end when the specified time interval has elapsed. The averaged data point will be shown as displayed in Figure 92. The **SELECT DISP** key can be used to view the average value of all measurement parameters. The **PRINT** key can be used to send a single averaged dataline to the printer.

```
   DATA AVERAGING
  300 READINGS WERE
 AVG'D OVER 02:30 M:S
 AVERAGE TORQUE=90.3
```

Figure 92

> **Note:** Printing during a Timed Average test will show non-averaged data. Averaged data is available only at the conclusion of the test. Printing the averaged data must be done with the **PRINT** key at the end of the test.

APPENDIX A - Cone/Plate Rheometer Set-Up

This Cone/Plate version of the DV-III Ultra uses the same operating instruction procedures as described in this manual. However, the "gap" between the cone and the plate must be verified/ adjusted before measurements are made. This is done by moving the plate (built into the sample cup) up towards the cone until the pin in the center of the cone touches the surface of the plate, and then by separating (lowering) the plate 0.0005 inch (0.013mm).

Programmable DV-III Ultra Cone/Plate Viscometers, S/N 50969 and higher, have an Electronic Gap Setting feature. This feature enables the user to easily find the 0.0005 inch gap setting that was established at Brookfield prior to shipment.

The following information explains how to set the Electronic Gap and verify calibration of the DV-III Ultra Viscometer.

A.1 ELECTRONIC GAP SETTING FEATURES

TOGGLE SWITCH allows you to enable/disable the Electronic Gap Setting Feature: left position is OFF (disabled), right position is ON (enabled).

PILOT LIGHT is the red (LED) light; when illuminated, it means the Electronic Setting Function is sensing (enabled).

CONTACT LIGHT is the yellow (LED) light; when it first turns on, the "hit point" has been found.

SLIDING REFERENCE MARKER is used after finding the "hit point;" it is the reference for establishing the 0.0005 inch gap.

MICROMETER ADJUSTMENT RING is used to move the cup up or down in relation to the cone spindle. Turning the ring left (clockwise) lowers the cup; turning it right (counterclockwise) raises the cup. Each line on the ring represents one scale division and is equivalent to 0.0005 inch movement of the plate relative to the cone.

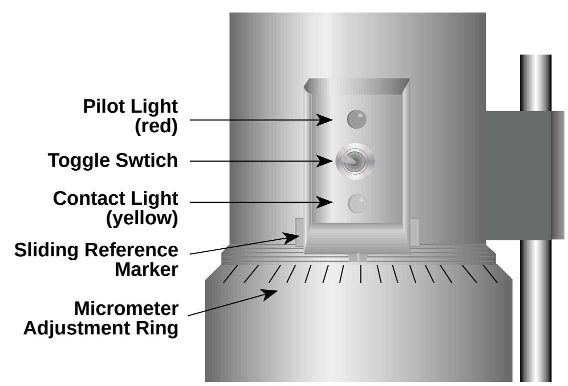


Figure A1

A.2 SETUP

1. Be sure that the Viscometer is securely mounted to the Laboratory Stand, leveled and zeroed with no cone or cup attached and 0% torque is displayed.

2. **Figure A2** shows a typical water bath setup. Connect the sample cup inlet/outlet ports to the water bath inlet and outlet and set the bath to the desired test temperature. Allow sufficient time for the bath to reach the test temperature.

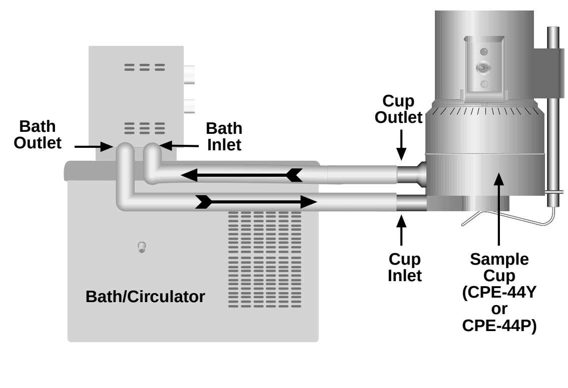


Figure A2

3. The Viscometer has been supplied with a special cone spindle(s) which contains the Electronic Gap Setting feature. The "CPE" part number designation on the cone verifies the Electronic Gap Setting feature. **Note**: The "CPE" cone or cup cannot be used with earlier DV-III Ultra cone/plate Viscometers (below S/N50969) which do not have the electronic gap setting feature.

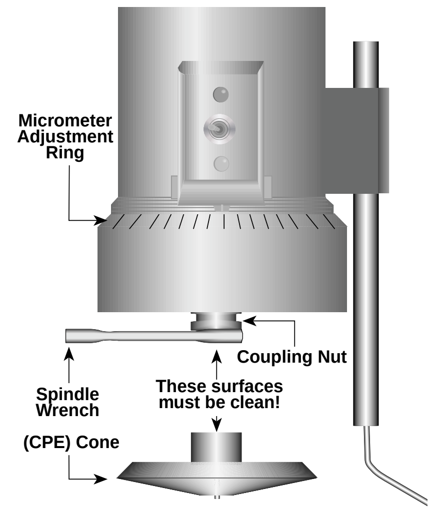


Figure A3

4. With the motor off, thread the cone spindle by using the spindle wrench to secure the viscometer coupling nut (see **Figure A3**); gently push up on the coupling nut and hold this securely with the wrench. Thread the cone spindle by hand. **Note:** <u>Left Hand Threads.</u>

5. Attach the cup, taking care not to hit the cone with the cup (**Figure A4**).

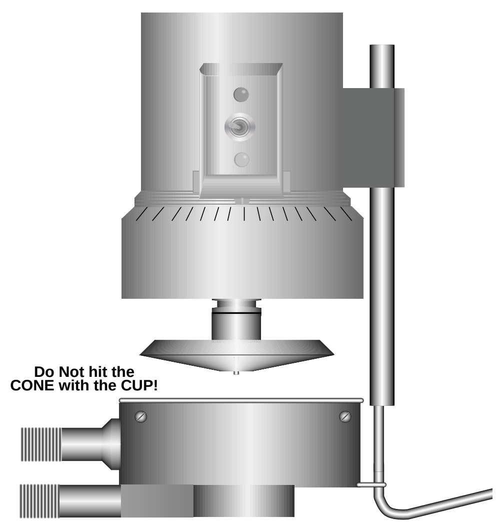


Figure A4

> The viscosity of electrically conductive fluids may be affected if readings are taken with the Electronic Gap Setting feature "on". Be sure to shut the feature "off" before taking readings!

A.3 SETTING THE GAP

1. Move the toggle switch to the right; this will turn on (enable) the Gap Setting Feature. The Pilot (red) light will be illuminated.

2. If the contact light (yellow) is illuminated, turn the micrometer adjustment ring clockwise (as you look down on the instrument) until the light is just breaking contact, i.e., flickering (see **Figure A5**).

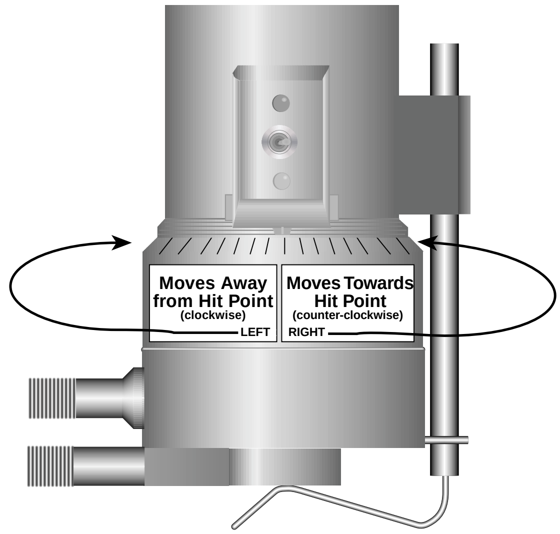


Figure A5

3. If the yellow contact light is not illuminated, *slowly* turn the micrometer adjustment ring in small increments (one or two scale divisions) counter-clockwise.

   Continue moving the micrometer adjustment ring *slowly* counter-clockwise until the contact light (yellow) turns on. Back off (rotate clockwise) until the light is just breaking contact, i.e., flickering.

4. Adjust the sliding reference marker, right or left, to the closest full scale division mark **(see Figure A6)**.

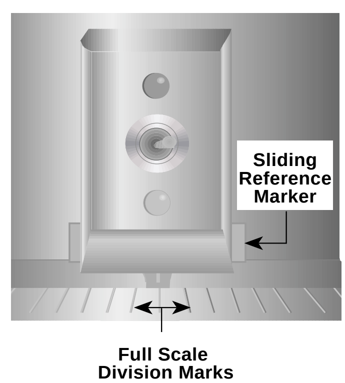


Figure A6

5. Turn the micrometer adjustment ring one scale division to the left to meet the line on the sliding reference marker. **THE YELLOW CONTACT LIGHT SHOULD GO OFF**.

6. You have established the gap space needed for measurement. Now turn the toggle switch OFF (left); the red pilot light should go off.

7. Carefully remove the sample cup.

Notes
1. The cup may be removed and replaced without resetting the gap if the micrometer adjustment ring has not been moved.
2. Remove the spindle from the viscometer when cleaning.
3. Re-establish the hit point every time the spindle is attached/detached.

A.4 VERIFYING CALIBRATION

1. Determine the appropriate sample volume. Refer to Table A1 to determine the correct sample volume required for the spindle to be utilized.

2. Select a Brookfield Viscosity Standard fluid that will give viscosity readings between 10% and 100% of full scale range. Refer to **Appendix B** for viscosity ranges of cone spindles; ranges listed apply to CPE cones.

   Do not use a silicone viscosity standard fluid with a viscosity value greater than 5000 cP with a Cone/Plate. Brookfield offers a complete range of mineral oil viscosity standards suitable for use with Cone/Plates for viscosities above 5,000 cP or shear rates above 500 sec^{-1}; see Table E2 in Appendix E for a list of available fluids.

   It is best to use a viscosity standard fluid that will be close to the maximum viscosity for a given cone spindle/speed combination.

   Example: LVDV-III Ultra Viscometer, Cone Spindle CPE-42, Brookfield Silicone Viscosity Standard having a viscosity of 9.7 cP at 25°C

   At 60 RPM, the full scale viscosity range is 10.0 cP. Thus, the Viscometer reading should be 97% torque and 9.7 cP viscosity ± 0.197 cP. The allowable error (±0.197 cP) is a combination of Viscometer accuracy and fluid tolerance (refer to **Interpretation of Calibration Test Results** in Appendix E).

3. Set the gap as described in Section A.3.

4. With the motor off, remove the sample cup and place the viscosity standard fluid into the cup.

Table A6

Cone Part No.	Sample Volume
CPE-40	0.5 ml
CPE-41	2.0 ml
CPE-42	1.0 ml
CPE-51	0.5 ml
CPE-52	0.5 ml

Table A1

5. Attach the sample cup to the Viscometer and allow sufficient time for the sample, cup and cone to reach temperature equilibrium.

6. Turn the motor on. Set the desired speed(s). Measure the viscosity and record the reading in both % torque and centipoise (cP).

   Note: The cone spindle must rotate at least five (5) times before a viscosity reading is taken.

7. Verify that the viscosity reading is within the allowable 1% deviation, as explained earlier, for the specific viscosity standard fluid(s) that you are using.

* The CPE designation on the cone spindle indicates use with Electronic Gap Setting Cone/Plate Viscometers/ Rheometers **only**.

APPENDIX B - Viscosity Ranges

The table below (Universal Spindle Ranges) lists the Spindle Range Coefficients for all spindles used on DV-III Ultra Rheometers. Dividing the coefficient number by any of the 2,500 Rheometer speeds will give the full scale viscosity range for a Rheometer/spindle/speed combination.

Universal Spindle Ranges

Spindle	Entry Code	SPINDLE RANGE COEFFICIENT Rheometer Series			
		LV	RV	HA	HB
RV1	01	937	10,000	20,000	80,000
RV2	02	3,750	40,000	80,000	320,000
RV3	03	9,375	100,000	200,000	800,000
RV4	04	18,750	200,000	400,000	1,600,000
RV5	05	37,500	400,000	800,000	3,200,000
RV6	06	93,750	1,000,000	2,000,000	8,000,000
RV7	07	375,000	4,000,000	8,000,000	32,000,000
HA1	01	937	10,000	20,000	80,000
HA2	02	3,750	40,000	80,000	320,000
HA3	03	9,375	100,000	200,000	800,000
HA4	04	18,750	200,000	400,000	1,600,000
HA5	05	37,500	400,000	800,000	3,200,000
HA6	06	93,750	1,000,000	2,000,000	8,000,000
HA7	07	375,000	4,000,000	8,000,000	32,000,000
HB1	01	937	10,000	20,000	80,000
HB2	02	3,750	40,000	80,000	320,000
HB3	03	9,375	100,000	200,000	800,000
HB4	04	18,750	200,000	400,000	1,600,000
HB5	05	37,500	400,000	800,000	3,200,000
HB6	06	93,750	1,000,000	2,000,000	8,000,000
HB7	07	375,000	4,000,000	8,000,000	32,000,000
LV1	61	6,000	64,000	128,000	512,000
LV2	62	30,000	320,000	640,000	2,560,000
LV3	63	120,000	1,280,000	2,560,000	10,240,000
LV4	64	600,000	6,400,000	12,800,000	51,200,000
LV5	65	1,200,000	12,800,000	25,600,000	102,400,000
T-A	91	18,750	200,000	400,000	1,600,000
T-B	92	37,440	400,000	800,000	3,200,000
T-C	93	93,600	1,000,000	2,000,000	8,000,000
T-D	94	187,200	2,000,000	4,000,000	16,000,000
T-E	95	468,000	5,000,000	10,000,000	40,000,000
T-F	96	936,000	10,000,000	20,000,000	80,000,000
Spiral	70	98,400	1,050,000	2,100,000	8,400,000
ULA	00	600	6,400	12,800	51,200

Universal Spindle Ranges

Spindle	Entry Code	SPINDLE RANGE COEFFICIENT Rheometer Series			
		LV	RV	HA	HB
DIN-81	81	3,420	36,500	73,000	292,000
DIN-82	82	3,420	36,500	73,000	292,000
DIN-83	83	11,340	121,300	242,600	970,400
DIN-85	85	1,144	12,200	24,400	97,600
DIN-86	86	3,420	36,500	73,000	292,000
SC4-14	14	117,200	1,250,000	2,500,000	10,000,000
SC4-15	15	46,880	500,000	1,000,000	4,000,000
SC4-16	16	120,000	1,280,000	2,560,000	10,240,000
SC4-18	18	3,000	32,000	64,000	256,000
SC4-21	21	4,688	50,000	100,000	400,000
SC4-25	25	480,000	5,120,000	10,240,000	40,960,000
SC4-27	27	23,4000	250,000	500,000	2,000,000
SC4-28	28	46,8880	500,000	1,000,000	4,000,000
SC4-29	29	93,750	1,000,000	2,000,000	8,000,000
SC4-31	31	30,000	320,000	640,000	2,560,000
SC4-34	34	60,000	640,000	1,280,000	5,120,000
SC4-37	37	23,4000	250,000	500,000	2,000,000
CP-40	40	307	3,270	6,540	26,160
CP-41	41	1,151	12,280	24,560	98,240
CP-42	42	600	6,400	12,800	51,200
CP-51	51	4,800	51,200	102,400	409,600
CP-52	52	9,216	98,300	196,600	786,400
V-71	71	2,456	26,200	52,400	209,600
V-72	72	10,404	111,000	222,000	888,000
V-73	73	50,146	535,000	1,070,000	4,280,000
V-74	74	508,954	5,430,000	10,860,000	43,440,000

Notes: RV spindle ranges are calculated with the RV Guardleg in use.
LV spindle ranges are calculated with the LV Guardleg in use.

Example 1: Determine the full scale viscosity range of the LV3 spindle running on an RV series Rheometer at 45 RPM.
Spindle Speed = 45 RPM
LV Spindle Range Coefficient for RV series Rheometer = 1,280,000
Full Scale Viscosity Range = $\frac{1,280,000}{45}$ = 28,444 cP (mPa•s)

Example 2: Determine the full scale viscosity range of the LV3 spindle running on a 2xHA Rheometer at 103.5 RPM.
Spindle Speed = 103.5 RPM
LV3 Spindle Range Cooefficient for 2xHA Rheometer = 2 x 2,560,000
Full Scale Viscosity Rnage = $\frac{5,120,000}{103.5}$ = 49,468 cP (mPa•s)

Note: The maximum viscosity taht should be taken from the DV-III Ultra Rheometer is at 100% of any full scale spindle/speed range.

LV(#1-4) and RV,HA,HB(#1-7) Rheometers

Viscosity Range (cP)		
Viscometer	**Minimum**	**Maximum**
LVDV-III Ultra	15	6,000,000
RVDV-III Ultra	100	40,000,000
HADV-III Ultra	200	80,000,000
HBDV-III Ultra	800	320,000,000

Small Sample Adapter

Spindle	Shear Rate (sec-1)	Viscosity (cP)			
		LVDV-III Ultra	RVDV-III Ultra	HADV-III Ultra	HBDV-III Ultra
14	0 - 80	47.0 - 1,171,000	500.0 - 12,500,000	1,000.0 - 25,000,000	4,000 - 100,000,000
15	0 - 96	19.0 - 468,650	200.0 - 5,000,000	400.0 - 10,000,000	1,600 - 40,000,000
16	0 - 58	48.0 - 1,199,700	512.0 - 12,800,000	1,024.0 - 25,600,000	4,100 - 102,400,000
18	0 - 264	1.3 - 30,000	12.8 - 320,000	25.6 - 640,000	103 - 2,560,000
21	0 - 186	1.9 - 46,865	20.0 - 500,000	40.0 - 1,000,000	160 - 4,000,000
25	0 - 44	192.0 - 4,790,000	2,050.0 - 51,200,000	4,100.0 -102,400,000	16,400 - 409,600,000
27	0 - 68	9.4 - 234,325	100.0 - 2,500,000	200.0 - 5,000,000	800 - 20,000,000
28	0 - 56	18.8 - 468,650	200.0 - 5,000,000	400.0 - 10,000,000	1,600 - 40,000,000
29	0 - 50	37.5 - 937,300	400.0 - 10,000,000	800.0 - 20,000,000	3,200 - 80,000,000
31	0 - 68	12.0 - 300,000	128.0 - 3,200,000	256.0 - 6,400,000	1,024 - 25,600,000
34	0 - 56	24.0 - 600,000	256.0 - 6,400,000	512.0 - 12,800,000	2,048 - 51,200,000
37	0 - 72	9.4 - 234,325	100.0 - 2,500,000	200.0 - 5,000,000	800 - 20,000,000
82	0 - 258	1.4 - 10,000	15.0 - 10,000	30.0 - 10,000	120 - 10,000
83	0 - 258	4.6 - 50,000	50.0 - 50,000	100.0 - 50,000	400 - 50,000

Thermosel

Spindle	Shear Rate (sec-1)	Viscosity (cP)			
		LVDV-III Ultra	RVDV-III Ultra	HADV-III Ultra	HBDV-III Ultra
18	0 - 264	1.3 - 30,000	12.8 - 320,000	25.6 - 640,000	103 - 2,560,000
21	0 - 186	1.9 - 46,865	20.0 - 500,000	40.0 - 1,000,000	160 - 4,000,000
27	0 - 68	9.4 - 234,325	100.0 - 2,500,000	200.0 - 5,000,000	800 - 20,000,000
28	0 - 56	18.8 - 468,650	200.0 - 5,000,000	400.0 - 10,000,000	1,600 - 40,000,000
29	0 - 50	37.5 - 937,300	400.0 - 10,000,000	800.0 - 20,000,000	3,200 - 80,000,000
31	0 - 68	12.0 - 300,000	128.0 - 3,200,000	256.0 - 6,400,000	1,024 - 25,600,000
34	0 - 56	24.0 - 600,000	256.0 - 6,400,000	512.0 - 12,800,000	2,048 - 51,200,000
81	0 - 258	1.4 - 10,000	15.0 - 10,000	30.0 - 10,000	120 - 10,000

UL Adapter

UL Spindle	Shear Rate (sec-1)	Viscosity (cP)			
		LVDV-III Ultra	RVDV-III Ultra	HADV-III Ultra	HBDV-III Ultra
YULA-15 or 15Z	0 - 122.4	1.0 - 2,000	3.2 - 2,000	6.4 - 2,000	25.6 - 2,000

Note: LV DV-III Ultra - Minimum viscosity, 1.0 cP at 60RPM, 10% of full scale range.

Note: RV,HA,HB-III - Minimum viscosity; 3 cP, 6 cP and 24 cP, at 230.9 RPM, 10% of full scale range.

Din Adapter

DAA Spindle	Shear Rate (sec-1)	Viscosity (cP) LVDV-III Ultra	RVDV-III Ultra	HADV-III Ultra	HBDV-III Ultra
85	0 - 258	0.6 - 5,000	6.1 - 5,000	12.2 - 5,000	48.8 - 5,000
86	0 - 258	1.8 - 10,000	18.2 - 10,000	36.5 - 10,000	146 - 10,000
87	0 - 258	5.7 - 50,000	61.0 - 50,000	121 - 50,000	485 - 50,000

Spiral Adapter

Spiral Spindle	Shear Rate (sec-1)	Viscosity (cP) LVDV-III Ultra	RVDV-III Ultra	HADV-III Ultra	HBDV-III Ultra
SA-70	.677 - 67.7 (1 - 100 RPM)	98.4 - 98,416	1,050 - 1,050,000	2,100 - 2,100,000	8,400 - 8,400,000

Cone/Plate Rheometer

Cone Spindle	Shear Rate (sec-1)	Viscosity (cP) LVDV-III Ultra C/P	RVDV-III Ultra C/P	HADV-III Ultra C/P	HBDV-III Ultra C/P
CPE-40	0 - 1500	0.15 - 3,065	1.7 - 32,700	3.3 - 65,400	13.1 - 261,000
CPE-41	0 - 400	0.58 - 11,510	6.2 - 122,800	12.3 - 245,600	49.1 - 982,400
CPE-42	0 - 760	0.3 - 6,000	3.2 - 64,000	6.4 - 128,000	25.6 - 512,000
CPE-51	0 - 768	2.4 - 47,990	25.6 - 512,000	51.2 - 1,024,000	205.0 - 4,096,000
CPE-52	0 - 400	4.6 - 92,130	49.2 - 983,000	98.3 - 1,966,000	393.0 - 7,864,000

Helipath with T-Bar Spindles

T-Bar Spindle	Viscosity (cP) LVDV-III Ultra	RVDV-III Ultra	HADV-III Ultra	HBDV-III Ultra
T-A	156 - 187,460	2,000 - 200,000	4,000 - 4,000,000	16,000 - 16,000,000
T-B	312 - 374,920	4,000 - 400,000	8,000 - 8,000,000	32,000 - 32,000,000
T-C	780 - 937,300	10,000 - 10,000,000	20,000 - 20,000,000	80,000 - 80,000,000
T-D	1,560 - 1,874,600	20,000 - 20,000,000	40,000 - 40,000,000	160,000 - 160,000,000
T-E	3,900 - 4,686,500	50,000 - 50,000,000	100,000 - 100,000,000	400,000 - 400,000,000
T-F	7,800 - 9,373,000	100,000 - 100,000,000	200,000 - 200,000,000	800,000 - 800,000,000

In taking viscosity measurements with the DV-III Ultra Rheometer there are two considerations which pertain to the low viscosity limit of effective measurement.

1) Viscosity measurements should be accepted within the equivalent % Torque Range from 10% to 100% for any spindle/speed combination.

2) Viscosity measurements should be taken under laminar flow conditions, not under turbulent flow conditions.

The first consideration has to do with the precision of the instrument. All DV-III Ultra Rheometers have a full scale range precision of (+/-) 1% of any spindle/speed combination. We discourage taking readings below 10% of range because the potential viscosity error of (+/-) 1% is a relatively high number compared to the instrument reading.

The second consideration involves the mechanics of fluid flow. All rheological measurements of fluid flow properties should be made under laminar flow conditions. Laminar flow is flow wherein all particle movement is in layers directed by the shearing force. For rotational systems, this means all fluid movement must be circumferential. When the inertial forces on the fluid become too great, the fluid can break into turbulent flow wherein the movement of fluid particles becomes random, and the flow can not be analyzed with standard math models. This turbulence creates a falsely high Rheometer reading, with the degree of non-linear increase in reading being directly related to the degree of turbulence in the fluid.

For the following geometries, we have found that an approximate transition point to turbulent flow occurs:

1) No. 1 LV Spindle: 15 cP at 60 RPM
2) No. 1 RV Spindle: 100 cP at 50 RPM
3) UL Adapter: 0.85 cP at around 70 RPM
4) SC4-18/13R: 1.25 cP at around 240 RPM

Turbulent conditions will exist in these situations whenever the RPM/cP ratio exceeds the values listed above.

APPENDIX C - Variables in Viscosity Measurements

As with any instrument, there are variables that can affect a viscosity measurement. These variables may be related to the instrument (Rheometer) or the test fluid. Variables related to the test fluid deal with the rheological properties of the fluid, while instrument variables would include the Rheometer design and the spindle geometry system utilized.

RHEOLOGICAL PROPERTIES

Fluids have different rheological characteristics that can be described by Rheometer measurements. We can then work with these fluids to suit our lab or process conditions.

There are two categories of fluids:

Newtonian - These fluids have the same viscosity at different Shear Rates (different RPM's) and are called Newtonian over the Shear Rate range they are measured.

Non-Newtonian - These fluids have different viscosities at different shear rates (different RPM's). They fall into two groups:

1) Time Independent non-Newtonian
2) Time Dependent non-Newtonian

The time dependency is the time they are held at a given Shear Rate (RPM). They are non-Newtonian, and when you change the Rheometer spindle speed, you get a different viscosity.

Time Independent

Pseudoplastic - A pseudoplastic material displays a decrease in viscosity with an increase in shear rate, and is also known as "shear thinning". If you take Rheometer readings from a low to a high RPM and then back to the low RPM, and the readings fall upon themselves, the material is time independent pseudoplastic and shear thinning.

Time Dependent

Thixotropic - A thixotropic material has decreasing viscosity under constant shear rate. If you set a Rheometer at a constant speed, recording viscosity (cP) values over time and find that the viscosity (cP) values decrease with time, the material is thixotropic.

Brookfield publication, "*More Solutions to Sticky Problems*" includes a more detailed discussion of rheological properties and non-Newtonian behavior.

Rheometer Related Variables

Most fluid viscosities are found to be non-Newtonian. They are Shear Rate dependent on the measurement conditions. The specifications of the Rheometer spindle and chamber geometry will affect the viscosity readings. If one reading is taken at 25 rpm, and a second at 50 rpm, the two viscosity (**cP**) values produced will be different because the readings were made at different shear rates. The faster the spindle speed, the higher the shear rate.

The shear rate of a given measurement is determined by: the rotational speed of the spindle, the size and shape of the spindle, the size and shape of the container used, and therefore, the distance between the container wall and the spindle surface.

A repeatable viscosity test should control or specify the following:

1) Test temperature
2) Sample container size (or spindle/chamber geometry)
3) Sample volume
4) Rheometer model
5) Spindle used (if using LVDV-III Ultra(#1-4) or RVDV-III Ultra(#1-7) attach the guard leg)
6) Test speed or speeds (or the shear rate)
7) Length of time or number of spindle revolutions to record viscosity.

APPENDIX D1 - Spindle and Model Codes for Viscosity Tests

Each spindle has a two digit code which is entered using the SPDL key on the DV-III Ultra key pad. The entry code allows the DV-III Ultra to calculate Viscosity, Shear Rate and Shear Stress values.

Each spindle has two constants which are used in these calculations. The Spindle Multiplier Constant (SMC) used for viscosity calculations, and the Shear Rate Constant (SRC), used for shear rate and shear stress calculations. Note that where SRC = 0, no shear rate/shear stress calculations are done and the data displayed is zero (0) for these functions.

Table D1

SPINDLE	ENTRY CODE	SMC	SRC
RV1	01	1	0
RV2	02	4	0
RV3	03	10	0
RV4	04	20	0
RV5	05	40	0
RV6	06	100	0
RV7	07	400	0
HA1	01	1	0
HA2	02	4	0
HA3	03	10	0
HA4	04	20	0
HA5	05	40	0
HA6	06	100	0
HA7	07	400	0
HB1	01	1	0
HB2	02	4	0
HB3	03	10	0
HB4	04	20	0
HB5	05	40	0
HB6	06	100	0
HB7	07	400	0
LV1	61	6.4	0
LV2	62	32	0
LV3	63	128	0
LV4	64	640	0
LV5	65	1280	0
T-A	91	20	0
T-B	92	40	0
T-C	93	100	0
T-D	94	200	0
T-E	95	500	0
T-F	96	1000	0
ULA	00	0.64	1.223
SC4-14	14	125	0.4
SC4-15	15	50	0.48
SC4-16	16	128	0.2929
SC4-18	18	3.2	1.32
SC4-21	21	5	0.93
SC4-25	25	512	0.22

Table D1 (continued)

SPINDLE	ENTRY CODE	SMC	SRC
SC4-27	27	25	0.34
SC4-28	28	50	0.28
SC4-29	29	100	0.25
SC4-31	31	32	0.34
SC4-34	34	64	0.28
SC4-37	37	25	0.36
CP40	40	0.327	7.5
CP41	41	1.228	2
CP42	42	0.64	3.8
CP51	51	5.12	3.84
CP52	52	9.83	2
Spiral Adapter	70	105	0.677
Thermosel DIN spindle	81	3.7	1.29
SSA DIN spindle for 13R or 13RP chamber	82	3.75	1.29
SSA DIN spindle for 7R or 7RP chamber	83	12.09	1.29
ULA DIN spindle	85	1.22	1.29
ULA DIN spindle	86	3.65	1.29
ULA DIN spindle	87	12.13	1.29

Table D2 lists the model codes and spring torque constants for each Rheometer model.

Table D2

MODEL	TK	MODEL CODE ON DV-III ULTRA SCREEN
LVDV-III Ultra	0.09373	LV
2.5xLVDV-III Ultra	0.2343	4L
5xLVDV-III Ultra	0.4686	5L
1/4 RVDV-III Ultra	0.25	1R
1/2 RVDV-III Ultra	0.5	2R
RVDV-III Ultra	1	RV
HADV-III Ultra	2	HA
2xHADV-III Ultra	4	3A
2.5xHADV-III Ultra	5	4A
HBDV-III Ultra	8	HB
2xHBDV-III Ultra	16	3B
2.5xHBDV-III Ultra	20	4B
5xHBDV-III Ultra	40	5B

The full scale viscosity range for any DV-III Ultra model and spindle may be calculated using the equation:

where: $$\text{Full Scale Viscosity Range [cP]} = \text{TK} * \text{SMC} * \frac{10{,}000}{\text{RPM}}$$

TK = DV-III Ultra Torque Constant from Table D2
SMC = Spindle Multiplier Constant listed in Table D1

The Shear Rate calculation is:

$$\text{Shear Rate } ({}^{1}/_{\text{sec}}) = \text{SRC} * \text{RPM}$$

where:

SRC = Shear Rate Constant from Table D1

Using Non-standard spindles with DV-III Ultra and RHEOCALC Software

Spindle Entry 99 allows entry of spindle constants which the DV-III Ultra will use to calculate Viscosity, Shear Rate and Shear Stress for spindles in boundary conditions other than the 600ml beaker or specified chamber.

The spindles must conform to geometries that allow for mathematical calculations of Shear Rate and Shear Stress i.e. coaxial cylinder.

Two constants are required:

a) **SMC** (**S**pindle **M**ultiplier **C**onstant) which is used to calculate cP value.

b) **SRC** (**S**hear **R**ate **C**onstant) which is used to calculate Shear Rate and Shear Stress.

If SRC=0 (Example, LV #1-4, RV,HA,HB #1-7 spindles), Shear Rate and Shear Stress values are not calculated and can not be displayed.

The SMC value for a spindle may be calculated as follows:

a) For new spindle conditions you calculate the SMC using a Newtonian fluid of known viscosity (Brookfield Viscosity Standard). This is done in the container with the new dimensions at the controlled temperature specified for the viscosity standard fluid.

b) The new full scale viscosity range is calculated for a selected RPM:

$$\text{R1} = \frac{100\ \text{n}}{\text{Y}}$$

where:

R1 = The new full scale viscosity range
n = The viscosity (in cP) of the Newtonian fluid
Y = The Torque % reading at the selected RPM

c) The SMC Value is then calculated:

$$\text{SMC} = \frac{[\text{Full Scale Viscosity Range (cP)}] * [\text{Selected RPM}]}{\text{TK} * 10{,}000}$$

where:

TK = DV-III Ultra Torque Constant from Table D2

The SRC value may be calculated for cylindrical spindle geometry using the following equation:

$$\text{Shear Rate (1/sec)} = \frac{2\omega R_c^2 R_b^2}{\chi^2 [R_c^2 - R_b^2]}$$

where:

R_c = Radius of the container (in centimeters)
R_b = Radius of the spindle (in centimeters)
χ = Radius at which the shear rate is to be calculated (normally the same value as R_b; in centimeters)
ω = Angular velocity of the spindle (Rad/Sec)

$$\omega = \frac{2\pi}{60} * N$$

N = Spindle speed in RPM

SMC and SRC values are entered in RHEOCALC software. See the HELP file for details.

APPENDIX D2 - Spindle Codes and Speeds for Yield Tests

Each spindle has a two (2) digit code which is selected via the EZ-Yield™ software. The code allows the DV-III Ultra to calculate Yield Stress.

The immersion mark selected affects the stress calculations. Ensure the selected immersion mark reflects the mark in use.

Each spindle has a Yield Multiplier Constant (YMC) for stress calculations and a Spindle Multiplier Constant (SMC) for calibration checks (see Appendix E) as shown in Table D3. Spindle dimensions are also listed.

Spindle	Spindle Code	YMC	SMC	Vane Length inches	Vane Length cm	Vane Diameter inches	Vane Diameter cm
V-71	71	0.5	2.62	2.708	6.878	1.354	3.439
V-72	72	2.0	11.1	1.706	4.333	.853	2.167
V-73	73	10.0	53.5	.998	2.535	.499	1.267
V-74	74	100.0	543	.463	1.176	.232	.589

Table D-3

Note: If the secondary immersion mark is selected, the YMC value is doubled.

The full scale Yield Stress range for any DV-III Ultra model and spindle may be calculated using the equation:

Full Scale Yield Stress Range (Pa) = TK x YMC x 10

where:
TK = DV-III Ultra Torque Constant from Table D-2
YMC = Yield Multiplier Constant from Table D-3

The following speeds are available for yield tests.

Pre-Shear Speeds (RPM)	
Range	**Increment**
0.01 to 0.09	0.01
0.1 to 5.0	0.1
6, 10, 12, 20, 30, 50, 60, 100, 200	N/A

Zero Step Speeds (RPM)	
Range	**Increment**
0.01 to 0.09	0.01
0.1 to 0.5	0.1

Run Speeds (RPM)	
Range	**Increment**
0.01 to 0.09	0.01
0.1 to 5.0	0.1

Table D-4

The shear stress measurement range for the three standard (or supplied) vane spindles at each spring torque is as follows:

Spindle	Model Code on DV-III Ultra Screen	TK	Shear Stress Range (Pa)
V-71	LV	.09373	0.047-0.47
V-72	LV	.09373	0.19-1.9
V-73	LV	.09373	0.94-9.4
V-71	RV	1	0.5-5
V-72	RV	1	2-20
V-73	RV	1	10-100
V-71	HB	8	4-40
V-72	HB	8	16-160
V-73	HB	8	80-800
V-71	5xHB	40	20-200
V-72	5xHB	40	80-800
V-73	5xHB	40	400-4000

Table D-5

Calculations for Shear Stress in Yield Test

The following equations can be used to calculate the shear stress values after each packet of data is obtained from the DV-III Ultra.

$$\tau = \frac{TK \times YMC \times T}{10}$$

where:

τ = Yield Stress (Pascals)

TK = Model Torque Constant from Table D-2

YMC = Yield Multiplier Constant from Table D-3

T = % Torque reading

The following equations can be used to calculate the strain data after each packet of data is obtained from the DV-III Ultra:

$$\gamma = \theta_M - (S \times T)$$

where:

γ = Strain (rad)

θ_M = Angular rotation of motor shaft (rad)

S = Radial Spring Factor (rad/% torque)

T = % Torque reading (%)

$$\theta_M = \omega \times t \times \frac{2\pi}{60}$$

where:

θ_M	=	Angular distance of motor shaft	(rad)
ω	=	Rotational speed	(rpm)
t	=	time of test	(seconds)

$$S = \theta_{cal} \times (2\pi) \times 0.01$$

where:

S	=	Radial spring factor	(rad/%torque)
θ_{cal}	=	Spring windup angle	(revolutions)

$$\theta_{cal} = \frac{(V_{cal} \times t_{cal})}{60000}$$

where:

θ_{cal}	=	Spring windup angle	(revolutions)
V_{cal}	=	Calibration speed (fixed at 0.1 rpm)	(rpm)
t_{cal}	=	Calibration time (time for 0% to 100% spring wind up)	(milliseconds)

$$t_{cal} = \frac{bi}{b_{cal}} \times \frac{V}{V_{cal}} \times 100$$

where:

t_{cal}	=	Calibration time (time for 0% to 100% spring windup during calibration)	(milliseconds)
bi	=	Base increment	(milliseconds)
b_{cal}	=	base increment calibration torque	(%torque/base increment)
V	=	Speed	(rpm)
V_{cal}	=	Calibration speed (fixed at 0.1 rpm)	(rpm)

APPENDIX E - Calibration Procedures

The accuracy of the DV-III Ultra is verified using viscosity standard fluids which are available from Brookfield Engineering Laboratories or your local Brookfield agent. Viscosity standards are Newtonian, and therefore, have the same viscosity regardless of spindle speed (or shear rate). Viscosity standards, calibrated at 25°C, are shown in Table E1.

Container size: For Viscosity Standards < 30,000 cP, use a 600 ml Low Form Griffin Beaker having a working volume of 500 ml.

For Viscosity Standards ≥ 30,000 cP, use the fluid container.

Inside Diameter: 3.25"(8.25cm)
Height: 4.75"(12.1cm)

Note: Container may be larger, but may not be smaller.

Temperature: As stated on the fluid standard label: (+/-) 0.1°C

Conditions: The DV-III Ultra should be set according to the operating instructions. The water bath should be stabilized at test temperature. Rheometers with the letters “LV” or “RV” in the model designation should have the guard leg attached.

Table E1

Normal 25°C Standard Fluids Viscosity (cP)	Normal 25°C Standard Fluids Viscosity (cP)	High Temperature Standard Fluids Three Viscosity/Temperatures**
5	5,000	HT-30,000
10	12,500	HT-60,000
50	30,000	HT-100,000
100	60,000	
500	100,000	**25°C, 93.3°C, 149°C
1,000		Refer to Brookfield catalog for more information.

Table E2

MINERAL OIL VISCOSITY STANDARD FLUIDS

BEL Part No.	Viscosity (cP) 25°C
B31	31
B210	210
B750	750
B1400	1,400
B2000	2,000
B11000	11,000
B20000	20,000
B80000	80,000
B200000	200,000
B420000	420,000

Brookfield Viscosity Standard Fluid - General Information

We recommend that Brookfield Viscosity Standard Fluids be replaced on an annual basis, one year from date of initial use. These fluids are pure silicone and are not subject to change over time. However, exposure to outside contaminants through normal use requires replacement on an annual basis. Contamination may occur by the introduction of solvent, standard of different viscosity or other foreign material.

Viscosity Standard Fluids may be stored under normal laboratory conditions. Disposal should be in accordance with state, local and federal regulations as specified on the material safety data sheet.

Brookfield Engineering Laboratories does not recertify Viscosity Standard Fluids. We will issue duplicate copies of the Certificate of Calibration for any fluid within two years of the purchase date.

Brookfield Viscosity Standard Fluids are reusable provided they are not contaminated. Normal practice for usage in a 600 ml beaker is to return the material from the beaker back into the bottle. When using smaller volumes in accessories such as Small Sample Adapter, UL Adapter or Thermosel, the fluid is normally discarded.

Calibration Procedure for LV(#1-4) and RV,HA,HB(#1-7) Brookfield spindles:

1) Place the viscosity standard fluid (in the proper container) into the water bath.

2) Lower the DV-III Ultra into measurement position (with guard leg if LV or RV series Rheometer is used).

3) Attach the spindle to the Rheometer. If you are using a disk shaped spindle, avoid trapping air bubbles beneath the disk by first immersing the spindle at an angle, and then connecting it to the Rheometer.

4) The viscosity standard fluid, together with the spindle, should be immersed in the bath for a minimum of 1 hour, stirring the fluid periodically, prior to taking measurements.

5) After 1 hour, check the temperature of the viscosity standard fluid with an accurate thermometer.

6) If the fluid is at test temperature (+/- 0.1°**C** of the specified temperature, normally 25°**C**), measure the viscosity and record the Rheometer reading.

   Note: The spindle must rotate at least five (5) times before readings are taken.

7) The viscosity reading should equal the cP value on the viscosity fluid standard to within the combined accuracies of the Rheometer and the standard (as discussed in the section entitled, Interpretation of Calibration Test Results).

Calibration Procedure for a Small Sample Adapter

When a Small Sample Adapter is used, the water jacket is connected to the water bath and the water is stabilized at the proper temperature:

1) Put the proper amount of viscosity standard fluid into the sample chamber. The amount varies with each spindle/chamber combination. (Refer to the Small Sample Adapter instruction manual.)

2) Place the sample chamber into the water jacket.

3) Put the spindle into the test fluid and attach the extension link, coupling nut and free hanging spindle (or directly attach the solid shaft spindle) to the DV-III Ultra.

4) Allow 30 minutes for the viscosity standard, sample chamber and spindle to reach test temperature.

5) Measure the viscosity and record the Rheometer reading.

Note: The spindle must rotate at least five (5) times before a viscosity reading is taken.

Calibration Procedure for a Thermosel System

When a Thermosel System is used, the controller stabilizes the Thermo Container at the test temperature. A two-step process is recommended for the Thermosel:

A) Evaluate the calibration of the Viscometer alone according to the procedure outlined in Appendix E, "Calibration Procedure for LV (#1-4) and RV,HA,HB (#1-7) Brookfield spindles."

B) Evaluate the Viscometer with Thermosel according to the procedure listed below:

a) Put the proper amount of HT viscosity standard fluid into the HT-2 sample chamber. The amount varies with the spindle used. (Refer to the Thermosel instruction manual).

b) Place the sample chamber into the Thermo Container.

c) Put the spindle into the test fluid and attach the extension link, coupling nut and free hanging spindle (or directly attach the solid shaft spindle) to the DV-III Ultra.

d) Allow 30 minutes for the viscosity standard, sample chamber and spindle to reach test temperature.

e) Measure the viscosity and record the Rheometer reading.

Note: The spindle must rotate at least five (5) times before a viscosity reading is taken.

Calibration Procedure using UL or DIN UL Adapters

When a UL or DIN UL Adapter is used, the water bath is stabilized at the proper temperature:

1) Put the proper amount of viscosity standard fluid into the UL Tube. (Refer to the UL Adapter instruction manual).

2) Attach the spindle (with extension link and coupling nut) onto the DV-III Ultra.

3) Attach the tube to the mounting channel.

4) Lower the tube into the water bath reservoir, or if using the ULA-40Y water jacket, connect the inlet/outlets to the bath external circulating pump.

5) Allow 30 minutes for the viscosity standard, sample chamber and spindle to reach test temperature.

6) Measure the viscosity and record the Rheometer reading.

Note: The spindle must rotate at least five (5) times before a viscosity reading is taken.

Calibration Procedure using a Helipath Stand and T-Bar Spindles

When a Helipath Stand and T-Bar spindles are used:

Remove the T-bar spindle and select a standard LV(#1-4) or RV,HA,HB(#1-7) spindle. Follow the procedures for LV(#1-4) and RV,HA,HB (#1-7) Brookfield spindles outlined above.

T-Bar spindles should not be used for verifying calibration of the DV-III Ultra Rheometer.

Calibration Procedure for Spiral Adapter

1) Place the viscosity standard fluid (in the proper container) into the water bath.

2) Attach the spindle to the viscometer. Attach chamber (SA-1Y) and clamp to the viscometer.

3) Lower the DV-III Ultra into measurement position. Operate the viscometer at 50 or 60 RPM until the chamber is fully flooded.

4) The viscosity standard fluid, together with the spindle, should be immersed in the bath for a minimum of 1 hour, stirring the fluid periodically (operate at 50 or 60 RPM periodically), prior to taking measurements.

5) After 1 hour, check the temperature of the viscosity standard fluid with an accurate thermometer.

6) If the fluid is at test temperature (+/- 0.1°C of the specified temperature, normally 25°C), measure the viscosity and record the viscometer reading.

   Note: The spindle must rotate at least five (5) times for one minute, whichever is greater before readings are taken.

7) The viscosity reading should equal the cP value on the viscosity fluid standard to within the combined accuracies of the viscometer and the standard (as discussed in the section entitled, Interpretation of Calibration Test Results).

Calibration Procedure for Cone/Plate Viscometers:

1) Follow the above procedures for mechanically adjusting the setting of the cone spindle to the plate.

2) Refer to Appendix A, Table A1, and determine the correct sample volume required for the selected spindle.

3) Select a viscosity standard fluid that will give viscosity readings between 10% and 100% of full scale range. Refer to Appendix B for viscosity ranges of cone spindles. Consult with Brookfield or an authorized dealer to determine which fluid is appropriate.

   It is best to use a viscosity standard fluid that will be close to the maximum viscosity for a given cone spindle/speed combination.

   Example: LVDV-III Viscometer, Cone CP-42, Fluid 10
   Having a viscosity of 9.7 cP at 25°C

   At 60 RPM, the full scale viscosity range is 10.0 cP. Thus, the Rheometer reading should be 97% torque and 9.7 cP viscosity ± 0.197 cP (0.1 cP for the rheometer plus 0.97 cP for the fluid). The accuracy is a combination of Rheometer and fluid tolerance (refer to Interpretation of Calibration Test Results).

4) With the viscometer stopped, remove the sample cup and place the viscosity standard fluid into the cup, waiting 10 minutes for temperature equilibrium.

5) Connect the sample cup to the Rheometer. Allow sufficient time for temperature to reach equilibrium. Typically 15 minutes is the maximum time that you must wait. Less time is required if spindle and cup are already at test temperature.

6) Measure the viscosity and record the Rheometer reading in both % torque and centipoise (cP).

Notes: 1) The spindle must rotate at least five (5) times before a viscosity reading is taken.

2) The use of Brookfield Viscosity Standard fluids in the range of 5 cP to 5000 cP is recommended for cone/plate instruments. Please contact Brookfield Engineering Laboratories or an authorized dealer if your calibration procedure requires more viscous standards.

3) Select a viscosity standard fluid that will give viscosity readings between 10% and 100% of full scale range. Refer to Appendix B for viscosity ranges of cone spindles. Do not use a silicone viscosity standard fluid with a viscosity value greater than 5000 cP with a Cone/Plate Viscometer. Brookfield offers a complete range of mineral oil viscosity standards suitable for use with Cone/Plate Viscometers as shown in Table E2. Consult with Brookfield or an authorized dealer to determine which fluid is appropriate.

Interpretation of Calibration Test Results:

When verifying the calibration of the DV-III Ultra, the instrument and viscosity standard fluid error must be combined to calculate the total allowable error.

The DV-III Ultra is accurate to (+/-) 1% of any full scale spindle/speed viscosity range.

Brookfield Viscosity Standards Fluids are accurate to (+/-) 1% of their stated value.

Example: Calculate the acceptable range of viscosity using RVDV-III with RV-3 Spindle at 2 RPM; Brookfield Standard Fluid 12,500 with a viscosity of 12,257 cP at 25°C:

1) Calculate full scale viscosity range using the equation:

$$\text{Full Scale Viscosity Range [}\mathbf{cP}\text{]} = \mathbf{TK} * \mathbf{SMC} * \frac{\mathbf{10{,}000}}{\mathbf{RPM}}$$

Where:
TK = 1.0 from **Table D2**
SMC = 10 from **Table D1**

$$\text{Full Scale Viscosity Range } \frac{\mathbf{1 * 10 * 10{,}000}}{\mathbf{2}} = \mathbf{50{,}000\ cP}$$

The viscosity is accurate to (+/-) 500 **cP** (which is 1% of 50,000)

2) The viscosity standard fluid is 12,257 cP. Its accuracy is (+/-)1% of 12,257 or (+/-)122.57 cP.

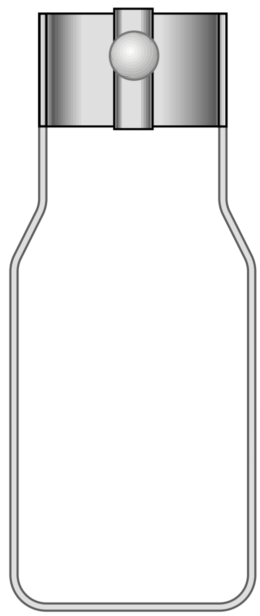
3) Total allowable error is (122.57 + 500) cP = (+/-) 622.57 cP.

4) Therefore, any viscosity reading between 11,634.4 and 12,879.6 cP indicates that the Rheometer is operating correctly. Any reading outside these limits may indicate a Rheometer problem. Contact the Brookfield technical sales department or your local Brookfield dealer/distributor with test results to determine the nature of the problem.

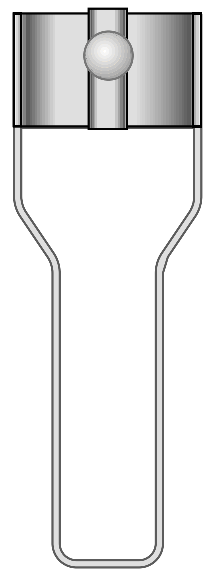
The Brookfield Guardleg

The *guard leg* was originally designed to protect the spindle during use. The first applications of the Brookfield Viscometer included hand held operation while measuring fluids in a 55 gallon drum. It is clear that under those conditions the potential for damage to the spindle was great. Original construction included a sleeve that protected the spindle from side impact. Early RV guard legs attached to the dial housing and LV guard legs attached to the bottom of the pivot cup with a twist and lock mechanism.

The current guard leg is a band of metal in the shape of the letter U with a bracket at the top that attaches to the pivot cup of a Brookfield Viscometer/Rheometer. Because it must attach to the pivot cup, the guard leg cannot be used with a Cone/Plate instrument. A guard leg is supplied with all LV and RV series instruments, but not with the HA or HB series. It's shape (shown right) is designed to accommodate the spindles of the appropriate spindle set; therefore, the RV guard leg is wider than the LV due to the large diameter of the RV #1 spindle. They are not interchangeable.



RV Guardleg



LV Guardleg

The calibration of the Brookfield Viscometer/ Rheometer is determined using a 600 ml Low Form Griffin Beaker. The calibration of LV and RV series instruments includes the guard leg. The beaker wall (for HA/HB instruments) or the guard leg (for LV/RV instruments) define what is called the "outer boundary" of the measurement. The spindle factors for the LV, RV, and HA/HB spindles were developed with the above boundary conditions. The spindle *factors* are used to convert the instrument torque (expressed as the dial reading or %Torque value) into centipoise. Theoretically, if measurements are made with different boundary conditions, e.g., without the guard leg or in a container other than 600 ml beaker, then the spindle factors found on the Factor Finder cannot be used to accurately calculate an absolute viscosity. Changing the boundary conditions does not change the viscosity of the fluid, but it does change how the instrument torque is converted to centipoise. Without changing the spindle factor to suit the new boundary conditions, the calculation from instrument torque to viscosity will be incorrect.

Practically speaking, the guard leg has the greatest effect when used with the #1 & #2 spindles of the LV and RV spindle sets. Any other LV (#3 & #4) or RV (#3 - #7) spindle can be used in a 600 ml beaker with or without the guard leg to produce correct results. The HA and HB series Viscometers/ Rheometers are not supplied with guard legs in order to reduce the potential problems when measuring high viscosity materials. HA/HB spindles #3 through #7 are identical to those spindle numbers in the RV spindle set. The HA/HB #1 & #2 have slightly different dimensions than the corresponding RV spindles. This dimensional difference allows the factors between the RV and HA/HB #1&#2 spindles to follow the same ratios as the instrument torque even though the boundary conditions are different.

The recommended procedures of using a 600 ml beaker and the guard leg are difficult for some customers to follow. The guard leg is one more item to clean. In some applications the 500 ml of test fluid required to immerse the spindles in a 600 ml beaker is not available. In practice, a smaller vessel may be used and the guard leg is removed. The Brookfield Viscometer/Rheometer will produce an accurate and repeatable torque reading under any measurement circumstance. However, the conversion of this torque reading to centipoise will only be correct if the factor used was developed for those specific conditions. Brookfield has outlined a method for recalibrating a Brookfield Viscometer/Rheometer to any measurement circumstance in More Solutions to Sticky Problems, Section 3.3.10. It is important to note that for many viscometer users the true viscosity is not as important as a repeatable day to day value. This repeatable value can be obtained without any special effort for any measurement circumstance. But, it should be known that this type of torque reading will not convert into a correct centipoise value when using a Brookfield factor if the boundary conditions are not those specified by Brookfield.

The guard leg is a part of the calibration check of the Brookfield LV and RV series Viscometer/Rheometer. Our customers should be aware of its existence, its purpose and the effect that it may have on data. With this knowledge, the viscometer user may make modifications to the recommended method of operation to suit their needs.

APPENDIX F - VS-27Y Clamp Assembly

The VS-35Y Clamp assembly holds the DV-III Ultra on the upright rod and thus supports it on the base. The parts are shown in **Figure F1**.

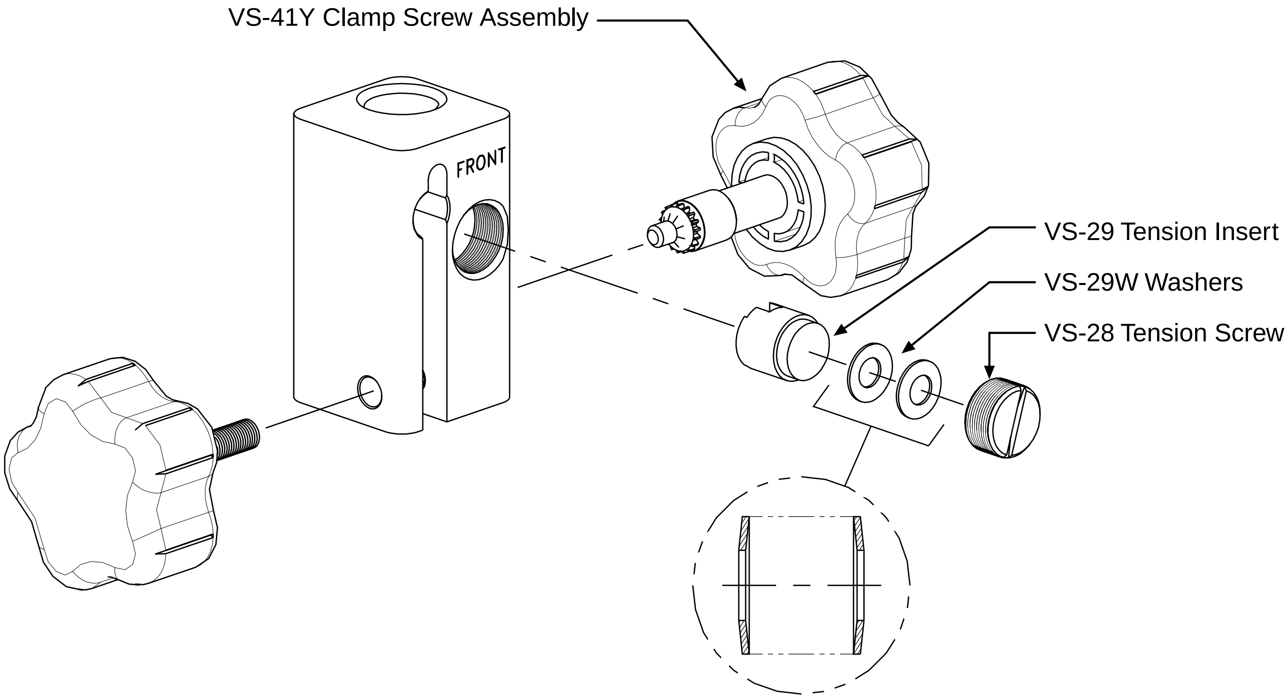


Figure F1

If the clamp is taken off the upright rod, the tension insert (Part No. VS-29) must be properly aligned for the clamp to fit back onto the upright rod.

When the tension insert (Part No. VS-29) is inserted, its slot must be in the vertical position parallel to the upright rod. If the slot is not in the correct position, the clamp will not slide down over the upright rod. Use a small screw driver or pencil to move it into the correct position. The VS-29W Belleville spring washers must face each other as illustrated. Adjust the VS-28 tension screw so that the clamp assembly is not loose on the upright rod.

APPENDIX G - DVE-50 Probe Clip

Probe Clip DVE-50 is supplied with all model DV-III Ultra Rheometers. It is used to attach the RTD temperature probe to a low form Griffin beaker. Figure G1 is a view of the Probe Clip, showing the hole into which the RTD probe is inserted. When inserting the RTD probe into the Probe Clip, the upper part of the Clip is compressed by squeezing the points shown in Figure G1.

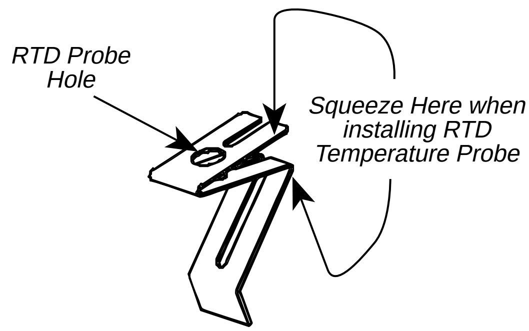


Figure G1

Figure G2 shows the probe clip mounted in a 600 ml low form Griffin beaker.

Note: The RTD probe must be parallel to the beaker wall so as not to interfere with the yield measurement.

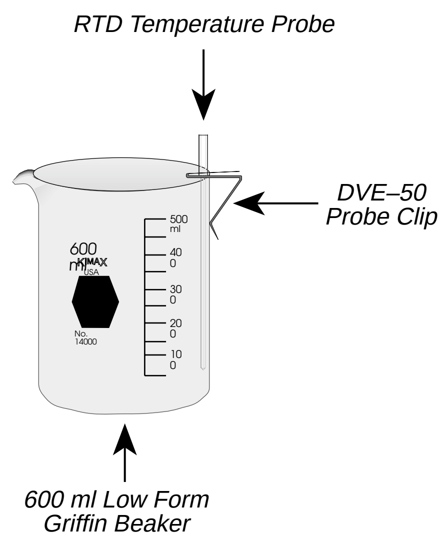
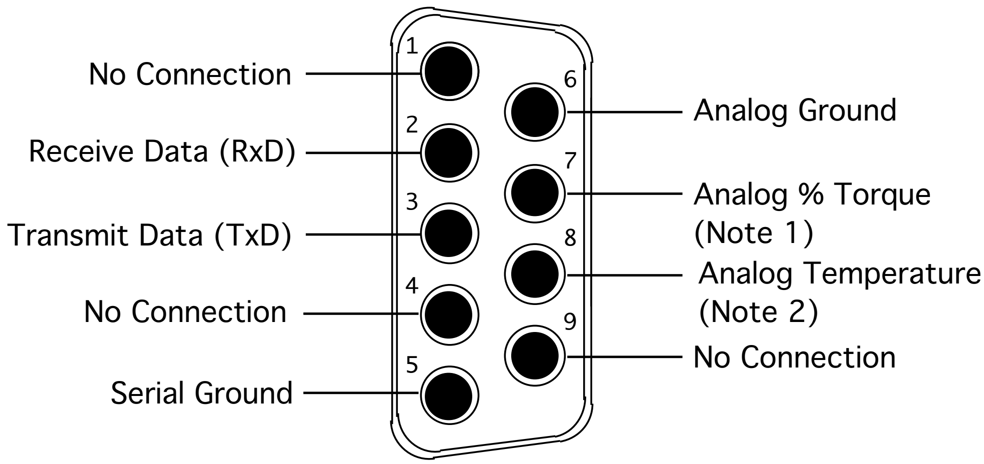


Figure G2

APPENDIX H - DV-III Ultra to Computer Command Set

Serial (RS-232) Communications Parameters

Baud Rate	9600
Data Bits	8
Stop Bits	1
Parity	None
Handshake	None



Notes:

1. This is a 0-1 volt d.c. output where 0 volts corresponds to 0% torque and 1 volt corresponds to 100 % torque with a resolution of 1 millivolt (0.1%).

2. This is a 0-4 volt d.c. output where 0 volts corresponds to -100 °C and 4 volts corresponds to +300 °C with a resolution of 1 millivolt (0.1°C).

Analog Output:

The analog outputs for temperature and % torque are accessed from the 9-pin connector located on the rear panel of the DV-III Ultra. The pin connections are shown in Figure E1. The output cable (Part No. DVP-96Y) connections are:

Red Wire:	Temperature Output
Black Wire:	Temperature Ground
White Wire:	% Torque Output
Green Wire:	% Torque GroundNote:

Please contact Brookfield Engineering Laboratories or your local dealer/distributor for purchase of the DVP-96Y analog output cable.

See Appendix D to perform the appropriate calculations using the data acquired during a test.

The command set used to communicate with the DV-III Ultra is as follows:

Command	Format from Host	Response from Rheometer	Description
E(nable)	<**E**><CR>	<**E**><ss><CR>	Enable control circuitry
R(etrieve)	<**R**><CR>	<**R**><qqqq><tttt><ss><CR>	Retrieve data
V(elocity)	<**V**><vvvvv><CR>	<**V**><ss><CR>	Send speed
I(dentify)	<**I**><CR>	<**I**><dddd><mm><xx><CR>	Identify instrument
Z(ero)	<**Z**><CR>	<**Z**><zzzz><ss><CR>	Zero instrument
Illegal String	<**????**><CR>	<**ss**><CR>	Invalid command

Where:

qqqq = Transducer reading as **4** hex digits. The instrument should yield a reading of approximately **0400H** after zeroing at rest. Calculate % torque as follows:

% **Torque** = **tval/100**

where **tval** is the decimal equivalent of the received torque packet.

zzzz = Transducer reading representing the % torque zero offset as **4** hex digits. Calculate the decimal equivalent of the zero offset as follows:

Zero offset = **tval/100**

where **tval** is the decimal equivalent of the received zero offset packet. This value should now be subtracted from all future % torque readings retrieved using the **R** command.

tttt = Temperature reading as **4** hex digits. Calculate temperature in °C as follows:

Temperature (°C) = (tval - 4000)/40

where **tval** is the decimal equivalent of the received temperature packet.

vvvvv = Stepper motor speed as **5** hex digits. The motor speed command from the Host is in units of **RPM**. To create a speed packet, multiply the desired decimal speed by 100 then convert the result to hexadecimal. All packets must be 5 characters long so for packets with less than that, pad the left side with zeros.

Example: To run the rheometer at 10 rpm

10 rpm x 100 = 1000 decimal = 3E8 hexadecimal;

padding with zeros results in 003E8 so the command sent would be V003E8

ss = Status Byte as **2** hex digits. (See Table 2)

dddd = The ASCII characters **"DV3+"**.

xx = The firmware version of the instrument (i.e. version 4.1 is returned as **41**)

mm = These **2** characters will be used to represent the model for which the **DV-III+** is configured. The model decoding is shown in **Table 1** below.

Model	mm
LV	LV
2.5LV	4L
5LV	5L
RV	RV
RV	1R
RV	2R
HA	HA
2HA	3A
2.5HA	4A
HB	HB
2HB	3B
2.5HB	4B
5HB	5B

Table 1 - DV-III Ultra Model Decoding

Invalid cmd received	1	x	x	x	x	x	x	x
Diagnostics error	x	x	1	x	x	x	x	x
Motor circuit error	x	x	x	1	x	x	x	x
Auto-zero complete	x	x	x	x	1	x	x	x
Motor speed = 0	x	x	x	x	x	1	x	x
Motor On (Energized)	x	x	x	x	x	x	1	x
Control circuitry enabled	x	x	x	x	x	x	x	1

Table 2 - Status Byte <ss> Definition

Note: These values are not updated in the status byte when the listed condition occurs. They are made available when the computer next sends a command that includes the status byte in the response. The flags are cleared when the condition causing a flag to be set has been resolved or by re-enabling (E command) the **DV-III Ultra**.

The **DV-III Ultra** must first be issued the **E**(nable) command to enable control circuitry and ascertain its current status. The **DV-III Ultra** will respond with an echo of the **E**(nable) command and will append the current status <**ss**> of the **DV-III Ultra**. This 2-digit status byte will provide information as to the rheometer's internal working condition and capability to continue with or to accept new tasks.

The **Z**(ero) command is used to "zero" the **DV-III Ultra** rheometer. The value returned <**zzzz**> is usually in the range of **03F0** hex to **0400** hex. This number should be retained and subtracted from <u>every</u> future returned torque reading to obtain the actual rheometer torque in percent.

The rheometer torque value and current temperature may be obtained by issuing the **R**(etrieve) command. The **DV-III Ultra's** response to this command is to echo the command: **R**(etrieve) and then follow it with **8** hex digits comprising the current values for the rheometer torque <**qqqq**> and the temperature probe reading <**tttt**>. Any control program written to exploit the output of the **DV-III Ultra** will have to perform all the calculations required for viscosity, shear stress and shear rate, etc. The **DV-III Ultra** will be directed to run at a given speed through the use of the **V**(elocity) command. Unlike the two previous commands, this command requires the parameter <**vvvvv**>

which contains the value for the desired speed. The **DV-III Ultra** responds to this command by repeating the command: **V**(elocity) and appending the status byte <**ss**> which will, amongst other things, inform the calling program as to whether the motor was turned on and/or whether the desired speed was attained.

RS-232 Output During The Yield Test

All output that occurs immediately before and after the yield test is described in Section III.4. That pre- and post- test output is sent out both the serial (RS-232) and parallel ports. When using the Brookfield Computer Cable (Brookfield part # DVP-80), the DV-III Ultra will output a data string during the actual yield test run. This data is ONLY sent out to the RS-232 port for use with the EZ-Yield™ software.

The following output is sent to the serial (RS-232) port at the end of every Base Increment period:

xxxxxx:yy.yy:ttt.t:zz.zz

where the delimiter between all fields is a colon (:) character and

xxxxxx	=	The Base Increment count in hexadecimal format, padded with leading zeroes. This is the time, in milliseconds that each reading was taken in the instrument.
yy.yy	=	% Torque value. % Torque is output with a resolution of 0.01% and a maximum of 99.99%.
ttt.t	=	Temperature in °C.
zz.zz	=	Delta Torque (%). This is the actual change in percent torque, measured from the previous reading, calculated at the end of each Base Increment period.

APPENDIX I - Fault Diagnosis and Troubleshooting

Listed are some of the more common problems that you may encounter while using your rheometer.

❑ Spindle Does Not Rotate

- ✓ Make sure the rheometer is plugged in.
- ✓ Check the voltage rating on your rheometer (115V or 220V); it must match the wall voltage.
- ✓ Make sure the motor is ON and the desired rpm is selected.

❑ Spindle Wobbles When Rotating or Looks Bent

- ✓ Make sure the spindle is tightened securely to the rheometer coupling.
- ✓ Check the straightness of all other spindles; replace if bent.
- ✓ Inspect rheometer coupling and spindle coupling mating areas and threads for dirt; clean threads on spindle coupling with a 3/56 left-hand tap.
- ✓ Inspect rheometer coupling threads for wear; if the threads are worn, the unit needs service (see Appendix I). Check to see if spindles rotate eccentrically or wobble. There is an allowable runout of 1/32-inch in each direction (1/16-inch total) when measured from the bottom of the spindle rotating in air.
- ✓ Check to see if the rheometer coupling appears bent; if so, the unit is in need of service (see Appendix J, "How to Return Your Rheometer").

If you continue to experience problems with your rheometer, follow this troubleshooting section to help isolate potential problems.

❑ Perform an Oscillation Check

This check verifies the mechanical condition of the shaft and bearing assembly in the rheometer. Auto zero the instrument according to directions in the manual. With the spindle removed, motor off and the display showing % torque perform the oscillation check as follows:

1) Manually lift the spindle nut and rotate counter clockwise until the display shows 20%-30% deflection.

2) Release the spindle nut.

3) The % display should return to 0.0% ±0.1%.

❑ Viscometer Will Not Return to Zero

✓ Rheometer is not level
- Adjust the laboratory stand

✓ Pivot point or jewel bearing faulty
- Perform calibration check
- Contact Brookfield Engineering Laboratories, Inc. or your Brookfield dealer for repair.

❑ Inaccurate Readings

✓ Verify spindle, speed and model selection.

✓ If % readings are under-range (less than 10%), the units display (%, cP, D/cm^2, 1/sec) will flash; change spindle and/or speed.

✓ "EEEE" on the digital display means the unit is over-range (greater than 100%); reduce speed and/or change spindle.

✓ Verify test parameters: temperature, container, volume, method. Refer to:
- "More Solutions to Sticky Problems", Section III
- DV-III Ultra Digital Rheometer Operating Instructions, Appendix C, "Variables in Viscosity Measurements."

✓ Perform a calibration check; follow the instructions in Appendix E.

✓ Verify tolerances are calculated correctly.

✓ Verify the calibration check procedures were followed exactly.

If the unit is found to be out of tolerance, the unit may be in need of service. See Appendix J for details on "How to Return Your Rheometer."

❑ Display Reading Will Not Stabilize

✓ Special characteristic of sample fluid. There is no problem with the rheometer.
- Refer to Appendix C

✓ Check for erratic spindle rotation
- Verify power supply
- Contact Brookfield Engineering Laboratories, Inc. or your Brookfield dealer for repair.

✓ Bent spindle or spindle coupling.
- Contact Brookfield Engineering Laboratories, Inc. or your Brookfield dealer for repair.

✓ Temperature fluctuation in sample fluid
- Use temperature bath control.

❑ No Recorder Response

✓ Be sure the rheometer is not at ZERO reading.

✓ Be sure the recorder is ON and not on STANDBY.

✓ Verify the range settings.

✓ Check cable leads for clean connection.

❑ Recorder Pen Moves in Wrong Direction

✓ Output polarity reversed
- Reverse leads

❑ Rheometer Will Not Communicate with PC

✓ Check the comm port setting and ensure the correct port on the PC is being utilized.

✓ Ensure that the rheometer has the appropriate communications cable (Communications) connected to it and that the opposite end of the cable is connected to a valid COM (serial) port on the host PC).

✓ Ensure that the rheometer is powered "ON".

✓ Ensure that the appropriate COM port is selected on the Main Toolbar of the EZ-Yield™ software.

✓ Ensure that "DV-III Ultra" is selected on the Main Toolbox of the EZ-Yield Software.

✓ If there is still erratic or no communication, check the following:
- Click the **START** button. Select "Settings" then "Control Panel". Double click the "System" icon. Select the "Device Manager" tab. Select "Ports", highlight the COM port in use and click the **PROPERTIES** button (or double-click the COM port in use). Click "Port Settings" then "Advanced".
- Ensure the check box labeled "Use FIFO buffers (requires 16550 compatible UART)" is checked. Ensure that the sliders for the "Receive Buffer" and the "Transmit Buffer" are both set all the way to the left (Low). Click the **OK** buttons to accept the changes and get back to the desktop.
- If there is still a communication problem, follow the above procedure, but this time, uncheck the box labeled "Use FIFO buffers (requires 16550 compatible UART)". Again, check the **OK** buttons to accept the changes.

✓ Check the interconnecting cable for proper installation.

✓ Contact Brookfield or an authorized dealer with the following information:

- When the communication error occurs
- The exact text displayed when the error occurs
- Computer hardware details including processor speed, RAM, network cards, modems, etc.
- Computer operating system
- Total number of COM ports
- List of Brookfield equipment attached to COM ports and their corresponding COM port number

APPENDIX J - Warranty Repair and Service

Warranty

Brookfield Viscometers are guaranteed for one year from date of purchase against defects in materials and workmanship. They are certified against primary viscosity standards traceable to the National Institute of Standards and Technology (NIST). The Viscometer must be returned to **Brookfield Engineering Laboratories, Inc.** or the Brookfield dealer from whom it was purchased for no charge warranty service. Transportation is at the purchaser's expense. The Viscometer should be shipped in its carrying case together with all spindles originally provided with the instrument.

For repair or service **in the United States,** return to:

Brookfield Engineering Laboratories, Inc.
11 Commerce Boulevard
Middleboro, MA 02346 U.S.A.

Telephone: (508) 946-6200 FAX: (508) 946-6262
e-mail: sales@brookfield engineering.com website: www.brookfieldengineering.com

For repair or service **outside the United States,** consult **Brookfield Engineering Laboratories, Inc.** or the dealer from whom you purchased the instrument.

For repair or service in the **United Kingdom,** return to:

Brookfield Viscometers Limited
1 Whitehall Estate
Flex Meadow
Pinnacles West
Harlow, Essex CM19 5TJ, United Kingdom

Telephone: (44) 27/945 1774 FAX: (44) 27/945 1775
e-mail: sales@brookfield.co.uk website: www.brookfield.co.uk

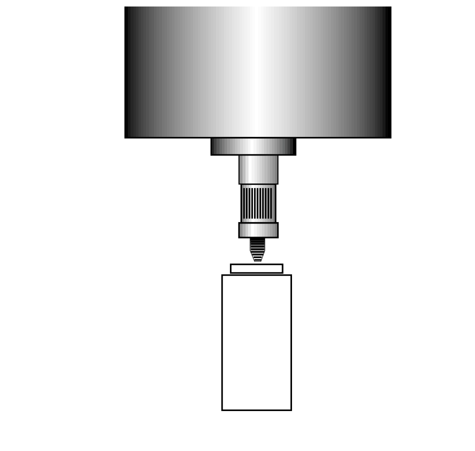
For repair or service in **Germany,** return to:

Brookfield Engineering Laboratories Vertriebs GmbH
Barbarossastrasse 3
D-73547 Lorch, Germany

Telephone: (49) 7172/927100 FAX: (49) 7172/927105
e-mail: info@brookfield-gmbh.de

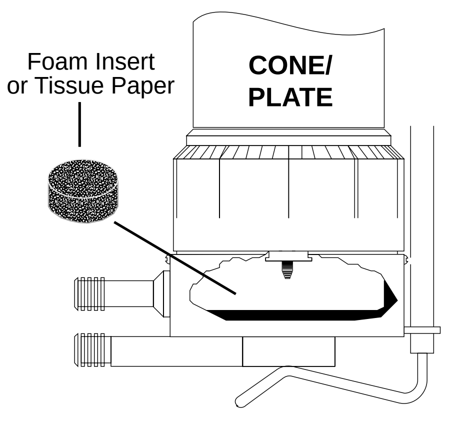
Packaging Instructions to Return a Viscometer for Repair or Calibration

FIGURE K1



- ❑ Remove and return all spindles (properly packed for shipping).
- ❑ Clean excess testing material off the instrument.
- ❑ Include MSDS sheets for all materials tested with this instrument.
- ❑ Support pointer shaft with white, nylon shipping cap, as shown in Figure K1, or with white plastic shipping cap originally supplied with instrument.
- ❑ Pack the instrument in its original case. Cases are available for immediate shipment from Brookfield. If the case is not available, take care to wrap the instrument with enough material to support it. Avoid using foam peanuts or shredded paper.
- ❑ DO NOT send the laboratory stand unless there is a problem with the upright rod, clamp or base. If there is a problem with the stand, remove the upright rod from the base and individually wrap each item to avoid contact with the instrument. Do not put lab stand in viscometer carrying case.

FIGURE K2



- ❑ Fill out a copy of the Viscometer Information Sheet (on the following page) with as much information as possible to help expedite your service *or* include a memo indicating the type of problem you are experiencing or the service you need performed. Please also include a purchase order number for us to bill against.
- ❑ Package the instrument and related items in a strong box for shipping. Mark the outside of the box with handling instructions.

  Example: "Handle with Care" or
  "Fragile - Delicate Instrument"

For cone/plate instruments, please remove the cone spindle and carefully pack in place in the shipping case. If available, use the original foam insert or roll up one sheet of tissue paper (or similar) and place between the spindle coupling and cup assembly (see Figure K2). This will help prevent damage in shipping.

Providing us with the following information will help us to service your equipment more quickly and efficiently. Please photocopy, fill out and return a copy of this form with your instrument.

Brookfield recommends that all viscometers be returned for annual calibration to ensure that your equipment continues to provide the same accuracy you have come to expect from Brookfield products.

1 VISCOMETER INFORMATION

Date: ________________________

Serial Number: ________________________ **Model:** ______________________

2 COMPANY INFORMATION

Company: __________________________ Primary User: __________________________

Telephone: _________________________ Fax: ________________________________

P.O. Number: _______________________ (to cover repair and shipping)

Billing Address: _____________________ Shipping Address: ______________________

___________________________________ ___________________________________

___________________________________ ___________________________________

___________________________________ ___________________________________

Return Shipment Instructions: UPS ground ❑ UPS Next Day ❑ UPS 2nd Day ❑

Federal Express ❑ _________________________________

(Federal Express Account Number required)

3 SERVICE INFORMATION

Operating Conditions (Spindle; Speed; Viscosity Range; Temp. Control; Temperature of Sample):

____

____

Did you contacting Brookfield before returning this instrument? Y❑ N❑

If yes, whom did you contact? ____

Description/Symptoms of Present Problem/Malfunction (please list all):

____

____

____

Time Since Last Serviced (if known): ______ Before & After Calibration Check? Y❑ N❑

Other Comments: ____

____

____

STEPS:
- ❑ Return the Viscometer to the attention of the Repair Department at the address above.
- ❑ Package the Viscometer for shipment, as outlined on the previous page.
- ❑ Include a purchase order or purchase order number with this form.

This tear-off sheet is a typical example of recorded test data. Please photocopy and retain this template so that additional copies may be made as needed.

VISCOSITY TEST REPORT

DATE:

BY:

FOR:

TEST INFORMATION:

SAMPLE	MODEL	SPINDLE	RPM	DIAL READING % TORQUE	FACTOR	VISCOSITY cP	SHEAR RATE	TEMP °C	TIME	NOTES

CONCLUSIONS:

BROOKFIELD ENGINEERING LABORATORIES, INC. • 11 Commerce Boulevard • Middleboro, MA 02346 • TEL: 508-946-6200 or 800-628-8139 (USA ex MA) FAX: 508-946-6262 • www.brookfieldengineering.com