

	<b>Standard Operating Procedure Calibration Verification and Operation of the Brookfield DV1 Digital Viscometer</b>	<b>SOP Number D-803</b>	<b>Revision 2</b>
		<b>Effective Date</b> 04/25/22	<b>Page Page 1 of 7</b>
<b>Written by/ Date</b> Steph S 02/24/22	<b>Reviewed by/ Date</b> Jon 03/21/22	<b>Approved by/ Date</b> SS 03/22/22	
<b>Title: Analytical Development Scientist</b>	<b>Title: Analytical Development Manager</b>	<b>Title: QC Laboratory Director</b>	

## 1.0 Purpose

This procedure provides instruction for the calibration verification and operation of the Brookfield DV1 digital viscometer.

## 2.0 Scope

This procedure applies to the Brookfield DV1 digital viscometer in use at Ion Labs, Inc.

## 3.0 Responsibility

- 3.1 It is the responsibility of QC Laboratory personnel to comply with this SOP.
- 3.2 It is the responsibility of QC Laboratory Management to implement this procedure and to ensure that the procedure is being followed.
- 3.3 It is the responsibility of QC Laboratory Management to keep this procedure current with the latest Ion Labs practices.

## 4.0 Definitions

- 4.1 **QC** – Quality Control
- 4.2 **K** – Thousand
- 4.3 **M** – Million
- 4.4 **Full Scale Range** – A calculation of the maximum viscosity value that can be measured by a spindle and speed combination when used with the specific spring torque of the DV1 viscometer

## 5.0 References

- 5.1 Brookfield DV1 Manual No. M14-023

<b>Standard Operating Procedure Calibration and Operation of the Brookfield DV1 Digital Viscometer</b>	<b>SOP No D-803</b>	<b>Rev 2</b>	<b>Page 2 of 7</b>
--	-------------------------	------------------	------------------------

5.2 RPT-21-0032, Report, Change to Calibration Frequency for Brookfield Viscometer

## **6.0 Procedure**

### **6.1 Location and Setup**

6.1.1 See Brookfield Manual M14-023, Appendix H if assembly is required.

6.1.2 The viscometer requires a clean, level surface for proper operation.

6.1.3 The viscometer is powered up by pressing the switch located on the back of the head and underneath the power cord. Allow the unit to warm up for 10 minutes before Auto-zeroing.

6.1.4 Verify that the viscometer head is level by inspecting the bubble located below the front panel. The bubble should be centered in the circle without touching or crossing the circle.

6.1.4.1 If leveling is required, the level is adjusted by turning either of the front feet clockwise or counter clockwise as necessary. Once complete, confirm that the bubble is appropriately centered in the circle before prior to each test. Should the head require releveling, the head should be Auto-zeroed.

### **6.1.5 Auto-zero**

6.1.5.1 Proper Auto-zero function is performed at power up and requires that the viscometer be level and the spindle be removed from the coupling shaft. Any spindle couple or extension links used with accessory device should be removed from the coupling shaft.

6.1.5.2 Press the NEXT hot key to initiate the Auto-zero. Upon completion, the DV1 presents a reminder to re-attach the spindle.

6.1.5.3 Press the NEXT hot key to proceed.

## **7.0 Usage**

### **7.1 Key Functions**

7.1.1 Three **HOT KEYS** are located immediately below the display. The action executed by the hot key will be indicated on the display. The actions available will vary with each screen. The typical actions include:

7.1.1.1 **BACK**: Return to previous screen

7.1.1.2 **SELECT**: Accept the parameter entered/chosen

7.1.1.3 **HOME**: Return to Home screen

7.1.1.4 **SPINDLE**: Enter Spindle Selection screen

7.1.1.5 **SPEED**: Enter Speed Selection screen

7.1.1.6 **OPTIONS**: Enter Options screen

7.1.1.7 **PRINT**: Print test data

7.1.1.8 **NEXT**: Advance to the next screen

7.1.2 **MOTOR OFF**: This key is used to turn off the motor, stop spindle rotation and stop current test.

7.1.3 **MOTOR ON**: This key is used to turn on the motor, start spindle rotation and start current test.

7.1.4 **UP ARROW**: This key is used to Scroll UP through the available speed or spindle tables.

7.1.5 **DOWN ARROW**: This key is used to Scroll Down through the available speed or spindle tables.

### **7.2 Spindle Selection and attachment**

7.2.1 Review the specifications for the material being tested to get the spindle size and RPM required for the test.

7.2.2 With the motor off, gently lift up and hold the coupling nut with one hand then attach the spindle by gently twisting it with the other hand, screw the correct size spindle for the application to the adaptor. Spindles connect to the adaptor with reverse threading. Turn counter-clockwise to tighten and clockwise to loosen.

7.2.3 The spindle size should also be changed on the display by pressing the SPINDLE hotkey on the home screen. Using the up and down arrow keys match the spindle on the display to the one attached to the motor.

7.2.4 The following spindles are available:

<b>Spindle</b>	<b>Range<sup>1</sup>- cP(mPa*s)</b>
RV-1	100 – 20K
RV-2	100 – 80K
RV-3	100 – 200K
RV-4	200 – 400K
RV-5	400 – 800K
RV-6	1K – 2M
RV-7	4K – 8M
LV-1	640 -128K
LV-2	3.2K – 640K
LV-3	12.8K – 2.56M

<sup>1</sup>Based on standard speeds 0.5 – 100 RPM.

### 7.3 Setting speed

7.3.1 Speed is set by pressing the SPEED hot key on the home screen. Using the up and down arrow keys to scroll through the list of available speeds. The Full Scale Range for each speed/spindle combination is displayed on the screen. Choose the speed with the most appropriate Full Scale Range for the sample being tested.

<b>Standard Operating Procedure Calibration and Operation of the Brookfield DV1 Digital Viscometer</b>	<b>SOP No D-803</b>	<b>Rev 2</b>	<b>Page 5 of 7</b>
--	-------------------------	------------------	------------------------

#### 7.4 System Suitability

- 7.4.1 System suitability should be performed prior to the first measurement of the day.
- 7.4.2 Choose a viscosity standard with viscosity in the range 1000 cP – 5000 cP.
- 7.4.3 Using a calibrated thermometer, record the temperature of the viscosity standard in the log book.
- 7.4.4 Ensure that the standard certificate of analysis has a viscosity listed for the current temperature of the standard (within 1°C).
- 7.4.5 Select the RV-3 spindle and 20 rpm speed.
- 7.4.6 Measure the viscosity of the standard as outlined below, and record the viscosity and torque readings in the log book.
- 7.4.7 Ensure that the measured viscosity of the standard is within 5% of that listed on the certificate of analysis.
- 7.4.8 Ensure that the measured torque is within the range 10% - 100%.
- 7.4.9 In the log book, indicate whether system suitability passes or fails.
- 7.4.10 Enter the initials and date of the person performing system suitability.

#### 7.5 Viscosity measurement

- 7.5.1 For standardization purposes all samples should be run at room temperature 23°C ± 2.
- 7.5.2 After spindle is attached and speed is set, place the spindle into the sample making sure the spindle is not touching any sides or bottom of the vessel and is fully submerged to the notch on the spindle, press motor on button to start measurement.
- 7.5.3 Accurate viscosity measurement requires time for the measurement sensor to equilibrate. Slower speeds require a longer time. Viscosity should not be documented until values are stable.

<b>Standard Operating Procedure</b> <b>Calibration and Operation of the Brookfield DV1</b> <b>Digital Viscometer</b>	<b>SOP No</b> <b>D-803</b>	<b>Rev</b> <b>2</b>	<b>Page</b> <b>6 of 7</b>
--	-------------------------------	------------------------	------------------------------

7.6 Cleaning

7.6.1 After each sample run, the spindle must be cleaned immediately. Sample drying or hardening on the spindle may cause scratching.

**Note:** Take care not to drop, warp or dent the spindle. Changes in the geometry of the spindle will affect the measurements.

**8.0 Calibration Verification**

8.1 Calibration verification should be performed yearly.

8.2 Three standards with viscosities of about 5000 cP, 12500 cP, and 30000 cP are used for calibration verification.

8.3 Record the spindle, speed, and full scale range used for each standard.

8.4 The viscosity tolerance of the instrument is the full scale range divided by 100.

8.5 The viscosity tolerance of the standard is the actual viscosity stated on the certificate of analysis divided by 100.

8.6 The allowable error for each standard is the viscosity tolerance of the instrument plus the viscosity tolerance of the standard.

8.7 The acceptable measurement range for each standard is the actual viscosity stated on the certificate of analysis  $\pm$  the allowable error.

8.8 Viscosity standards should be equilibrated to  $25.0 \pm 0.1$  °C prior to taking a reading.

8.9 After temperature equilibration of the standard, measure the viscosity of the standard as outlined in Section 7.

8.10 The calibration verification is passing if all three standards are within the acceptable measurement range.

**9.0 Specifications: Model DV1MRV**

9.1 Spring Torque            7187.0 dyne\*cm                            0.7187 milli Newton\*m

9.2 Speeds (RPM)            0.5, 1, 2, 2.5, 4, 5, 10, 20, 50, 100

<b>Standard Operating Procedure</b> <b>Calibration and Operation of the Brookfield DV1</b> <b>Digital Viscometer</b>	<b>SOP No</b> <b>D-803</b>	<b>Rev</b> <b>2</b>	<b>Page</b> <b>7 of 7</b>
--	-------------------------------	------------------------	------------------------------

- 9.3 Viscosity Accuracy: +/- 1% of Full Scale Range in Use
- 9.4 Viscosity Repeatability: +/- 0.2% of Full Scale Range in Use
- 9.5 Temperature Accuracy: +/- 1°C: -100°C to 149°C
- 9.6 Operating Environment: 0°C to 40°C, 20% to 80% non-condensing atmosphere

## 10.0 Revision History

Revision	Date	Description of Changes	CCR #	By
0	06/23/16	New	16-0989	J. Maignan
1	04/14/20	Fixed typos and added language to aid in reproducibility	CC-20-0273	J. Maignan
2	02/24/22	Added instructions for calibration verification, added system suitability section, changed calibration verification to yearly.	CC-22-0081	S. Sassman