Ti-Pure®

TITANIUM DIOXIDE

DETERMINATION OF BROOKFIELD VISCOSITY, TiO₂ SLURRY

METHOD: T4400.535.01.WP
I. Principle

Viscosity is determined by measuring the torque required to rotate a spindle in a fluid, employing a calculating, non-calculating, or dial type Brookfield viscometer.

II. Applicability

This method is applicable for the measurement of viscosity of all rutile slurry grades in the range of 50-1000 centipoise.

Following the manufacturer's instructions, spindles and rotational speed different from those specified may be used with equal end results.

III. Limitations

Extreme temperatures deviations from ambient temperature can cause significant variation in viscosity. Random studies indicate that normal variation in viscosity ranges 15-20% over the temperature span of 20°-30°C. Temperature control is not critical unless results approach specification limits.

IV. Sensitivity, Precision and Accuracy

A. Sensitivity

The limit of detection for this method is estimated at 3 centipoise.
B. Precision

1. Single Operator

The average analysis ($\bar{X}$), standard deviation ($s$), and 95% confidence limits (95% CL) established for the single operator precision of this method were as follows:

\[
\begin{array}{ccc}
\text{RPS VANTAGE®}, \text{ cps} & 123.3 & 1.035 & \pm 0.904 \\
\end{array}
\]

The above data were calculated from eight replicate analyses of one sample performed by one technician over a period of time at the Edge Moor Control Laboratory.

2. Multiple Operator

The average analysis ($\bar{X}$), standard deviation ($s$), and 95% confidence limits (95% CL) established for the multiple operator precision of this method were as follows:

\[
\begin{array}{ccc}
\text{RPS VANTAGE®}, \text{ cps} & 124.5 & 3.132 & \pm 1.146 \\
\end{array}
\]

The above data were calculated from thirty-two replicate analyses of one slurry sample performed by four technicians over a period of time at the Edge Moor Control Laboratory.

C. Accuracy

Accuracy, claimed by Brookfield, is ± 1% of full scale range.

V. Special Apparatus (Equivalent apparatus may be substituted)

1. Brookfield viscometers: Either Model RVTDV-II (Calculating), Model RVTD (Non-calculating), or Model RVT (Dial Type); Brookfield Engineering Laboratories, Stoughton, MA.
2. Brookfield laboratory stand, Model B, Brookfield Engineering Laboratories, Stoughton, MA.

3. #3 Spindle, Brookfield Engineering Laboratories, Stoughton, MA.

4. #4 Spindle, Brookfield Engineering Laboratories, Stoughton, MA.

5. Thermometer, viscosity, Saybolt, calibrated 66° to 80°F, VWR, Cat. No. 61126-002 or calibrated 19° to 27°C, VWR, Cat. No. 61125-881.

VI. Reagents (Reagent grade except as noted)


   NOTE: It is recommended that a minimum of (2) test fluids be kept on hand for calibrating and checking the instrument. The fluids should be in the range of 100-500 centipoise and in the range of 1000 centipoises. Test fluid should be replaced on a yearly basis.

VII. Special Safety Considerations

A. Product Hazards

   No product hazards have been identified. However, Titanium Dioxide is regulated as an air contaminant and care should be exercised to minimize product dusting. Wipe up all spills with a wet paper towel. See MSDS #2816CR for more information on Titanium Dioxide.

B. Procedure Hazards

   None

VIII. Procedure

Separate procedures are provided below for each specific type of Brookfield viscometer.

For each viscometer model, follow the manufactures operating procedure.
Model RVTDV-II (Calculating, Digital Brookfield Viscometer)

A. Operating Conditions

1. Mount the viscometer securely on the Brookfield stand.

   NOTE: This instrument is very sensitive and should be handled as such. Any severe jars to the instrument can shift the alignment of the motor with the gear train and cause a binding or looseness which can interfere with the smooth rotation of the dial. Any internal damage to the spring, bearing, or pivot is not visible, but will be noticeable in the operation of the instrument.

2. Level the viscometer using the level adjustment knobs located on the base of the stand.

3. Autozero the viscometer as follows:

   - Detach any spindles from the viscometer shaft.

   NOTE: This is best done by lifting the shaft slightly with one hand and unscrewing the spindle (left-handed thread) with the other hand. Care should also be taken to avoid putting side thrusts on the shaft to protect its alignment.

   - Turn power switch "ON"
   - Turn motor switch "ON" and set selector speed to 10 rpm
   - Press "Autozero"
   - When the display stops flashing, turn motor switch "OFF"
   - Leave the power switch "ON"

4. The viscometer is now zeroed and in "Standby Mode".

B. Calibration

The viscometer is calibrated at 25 ± 1.0°C as follows:

1. Place the viscometer in "Standby Mode".

2. Fill a 600-mL low-form Griffin beaker at least 3/4 full of viscosity oil standard (certified by NIST or Brookfield) having a value in the desired working range. Lean the beaker slightly and pour the oil slowly down the inside wall to avoid bubble entrapment.
3. Allow the oil to reach 25 ± 1.0°C, then measure and record the temperature.

4. The viscometer should be calibrated with the spindle to be used, typically #3 or #4. Spindles which are dirty or damaged, i.e., scratched, bent, or nicked can give false readings. To setup the automatic calculation,

   - Press "SPDL" to enter the spindle number into the memory of the calculating viscometer; then,
   - Press "CPS" for direct readout in centipoise during measurement.

5. Insert the desired spindle into the beaker along the side of the beaker and move it to the center to remove any bubbles which may be trapped beneath the spindle.

6. Move the beaker position beneath the viscometer and attach the spindle so that the spindle remains submerged in the sample and is submerged to the groove on the spindle shaft.

   NOTE: Use care in attaching the spindle to avoid putting side thrusts on the shaft to protect its alignment. It is best to lift the shaft slightly with one hand while using the other hand to screw on the spindle which has a left-handed thread.

7. Measure viscosity as follows:

   - Turn motor "ON"
   - Set motor speed selector to "100 rpm"
   - Allow reading to stabilize
   - Record reading
   - Turn motor "OFF"
   - Repeat sequence for any additional measurements

8. The final reading should be within the individual site accepted tolerance of the certified value of the viscosity oil standard. If not, contact the manufacturer for repair instructions.

9. Remove and clean spindle prior to use for sample analysis.
C. Sampling

Samples are normally provided by production.

D. Sample Analysis

1. Place the viscometer in "Standby Mode".

2. Allow the sample bottle to reach room temperature and then shake vigorously to re-mix any settled solids in the slurry.

3. Fill a 600-mL, low-form Griffin beaker at least 3/4 full of slurry from the sample bottle.

   NOTE: Additional stirring may be necessary to eliminate any gross amounts of bubbles in the slurry after transfer to the beaker.

4. Select a #3 or #4 spindle assembly as follows:

   - For RPS VANTAGE® measurements, use #4 spindle.
   
   - For all other grades, use #3 spindle.

   Press "SPDL" and enter the number of the spindle, either "03" or "04" and press "CPS" for direct readout of results in centipoise.

5. Insert the appropriate spindle into the beaker along the side of the beaker and move it to the center to remove any bubbles which may be trapped beneath the spindle.

6. Move the beaker position beneath the viscometer and attach the spindle so that the spindle remains submerged in the sample and is submerged to the groove on the spindle shaft.

7. To measure slurry viscosity:

   - Turn the motor "ON"
   - Set motor speed selector to "100 rpm"
   - Allow reading to stabilize
   - Record reading in centipoise when stable
   - Repeat the sequence for additional measurements

8. Remove and clean the spindle before re-using for the next sample.
E. Calculations

1. The viscometer automatically calculates viscosity and provides a direct readout of sample viscosity in centipoise.

2. Report the viscosity in centipoise, the rpm, and the temperature of the sample at measurement in degrees centigrade.

Model RVTD (Non-Calculating, Digital Brookfield Viscometer)

(A) Operating Conditions

1. Mount the viscometer securely on the Brookfield stand.

   NOTE: This instrument is very sensitive and should be handled as such. Any severe jars to the instrument can shift the alignment of the motor with the gear train and cause a binding or looseness which can interfere with the smooth rotation of the dial. Any internal damage to the spring, bearing, or pivot is not visible, but will be noticeable in the operation of the instrument.

2. Level the viscometer by checking the bubble level and adjusting the leveling screws located on the legs of the stand.

3. Zero the viscometer as follows:

   - Detach any spindles from the viscometer shaft.

   NOTE: This is best done by lifting the shaft slightly with one hand and unscrewing the spindle (left-handed thread) with the other hand. Care should also be taken to avoid putting side thrusts on the shaft to protect its alignment.

   - Turn power switch "ON".
   - Turn motor switch "ON" and set speed selector to "10 rpm"
   - Turn the "ZERO" knob to set the display to "00.0"
   - Turn motor switch "OFF"
   - Leave power switch "ON"

4. The viscometer is now zeroed and in "Standby Mode".
(B) Calibration

The viscometer is calibrated at 25 ± 1.0°C as follows:

1. Place the viscometer in "Standby Mode".

2. Fill a 600-mL low-form Griffin beaker at least 3/4 full of viscosity oil standard (certified by NIST or Brookfield) having a value in the desired working range. Lean the beaker slightly and pour the oil slowly down the inside wall to avoid bubble entrapment.

3. Allow the oil to reach 25 ± 1.0°C, then measure and record the temperature, T.

4. The viscometer should be calibrated with the spindle to be used, typically #3 or #4. Spindles which are dirty or damaged, i.e., scratched, bent, or nicked can give false readings.

5. Insert the desired spindle into the beaker along the side of the beaker and move it to the center to remove any bubbles which may be trapped beneath the spindle.

6. Move the beaker position beneath the viscometer and attach the spindle so that the spindle remains submerged in the sample and is submerged to the groove on the spindle shaft.

   NOTE: Use care in attaching the spindle to avoid putting side thrusts on the shaft to protect its alignment. It is best to lift the shaft slightly with one hand while using the other hand to screw on the spindle which has a left-handed thread.

7. Measure viscosity as follows:

   - Turn motor "ON"
   - Set motor speed selector to "100 rpm"
   - Allow reading to stabilize, (10-15 seconds)
   - Record reading in centipoise when stable
   - Turn motor "OFF"
   - Repeat sequence for any additional measurements

8. Calculate viscosity in centipoise by multiplying the meter reading by the correct multiplication factor for the speed and the spindle. Obtain the correct factors using the factor finder slide rule provided with the viscometer.
The calculated viscosity should be within the individual site accepted tolerance of the certified value of the viscosity oil standard. If not, contact the manufacturer for repair instructions.

9. Remove and clean spindle prior to use for sample analysis.

(C) Sampling

Samples are normally provided by production.

(D) Sample Analysis

1. Place the viscometer in "Standby Mode".

2. Allow the sample bottle to reach room temperature and then shake vigorously to re-mix any settled solids in the slurry. Check and record the temperature of the sample.

3. Fill a 600-mL, low-form Griffin beaker at least 3/4 full of slurry from the sample bottle.

   NOTE: Additional stirring may be necessary to eliminate any gross amounts of bubbles in the slurry after transfer to the beaker.

4. Select a #3 or #4 spindle assembly as follows:

   - For RPS VANTAGE® measurements, use #4 spindle.
   - For all other grades, use #3 spindle.

5. Insert the appropriate spindle into the beaker along the side of the beaker and move it to the center to remove any bubbles which may be trapped beneath the spindle.

6. Move the beaker position beneath the viscometer and attach the spindle so that the spindle remains submerged in the sample and is submerged to the groove on the spindle shaft.
7. To measure slurry viscosity:

- Turn the motor "ON"
- Set motor speed selector to "100 rpm"
- Allow reading to stabilize. (10-15 seconds)
- Record reading in centipoise when stable

8. Remove and clean the spindle before re-using for the next sample.

(E) Calculations

1. Calculate viscosity in centipoise by multiplying the meter reading by the correct multiplication factor for the speed and the spindle. Obtain the correct factors using the factor finder slide rule provided with the viscometer.

2. Report viscosity in centipoise, the rpm, and the temperature of the sample at measurement.

Model RVT (Dial Type, Brookfield Viscometer)

(A) Operating Conditions

1. Mount the viscometer securely on the Brookfield stand.

   NOTE: This instrument is very sensitive and should be handled as such. Any severe jars to the instrument can shift the alignment of the motor with the gear train and cause a binding or looseness which can interfere with the smooth rotation of the dial. Any internal damage to the spring, bearing, or pivot is not visible, but will be noticeable in the operation of the instrument.

2. Level the viscometer by checking the bubble level and adjusting the leveling screws located on the legs of the stand and remove any spindles from the viscometer shaft.

   NOTE: This is best done by lifting the shaft slightly with one hand and unscrewing the spindle (left-handed thread) with the other hand. Care should also be taken to avoid putting side thrusts on the shaft to protect its alignment.
3. The viscometer is now in "Standby Mode".

(B) Calibration

The viscometer is calibrated at 25 ± 1.0°C as follows:

1. Place the viscometer in "Standby Mode".

2. Fill a 600-mL, low-form Griffin beaker at least 3/4 full of viscosity oil standard (certified by NIST or Brookfield) having a value in the desired working range. Lean the beaker slightly and pour the oil slowly down the inside wall to avoid bubble entrapment.

3. Allow the oil to reach 25 ± 1.0°C, then measure and record the temperature. Note: the calibration should be done as close to 25°C as possible.

4. The viscometer should be calibrated with the spindle to be used, typically #3 or #4. Spindles which are dirty or damaged, i.e., scratched, bent, or nicked can give false readings.

5. Insert the desired spindle into the beaker along the side of the beaker and move it to the center to remove any bubbles which may be trapped beneath the spindle.

6. Move the beaker beneath the viscometer and attach the spindle so that the spindle remains submerged in the sample and is submerged to the groove on the spindle shaft.

   NOTE: Use care in attaching the spindle to avoid putting side thrusts on the shaft to protect its alignment. It is best to lift the shaft slightly with one hand while using the other hand to screw on the spindle which has a left-handed thread.

7. Measure viscosity as follows:

   - Depress and hold the clutch (located on the back side of the meter), turn the motor switch "ON" (located on the right side of the meter), and set the motor speed selector to maximize the display reading for the expected viscosity.
- Release the clutch and allow the dial to rotate until the pointer stabilizes at a fixed position on the dial, usually 8-10 revolutions.

NOTE: At 100 rpm, this will take 10-15 seconds.

- Depress the clutch lever with a thumb and turn the motor "OFF" with the other hand, then read the position of the red pointer.

NOTE: If the pointer is not in view when the dial has come to rest, turn the motor switch "ON" and "OFF" rapidly until the pointer is visible in the window of the vision plate.

CAUTION: Always release the clutch while the spindle is still immersed, so that the pointer will float rather than snap back to "Zero".

- Take at least three readings, or more, if necessary, to obtain consistent readings. The readings should not have more than a 3% spread. If the spread is greater than 3%, more readings must be taken until 3 are recorded with no more than a 3% spread.

- Take an average of the three readings and record the value.

8. Calculate viscosity in centipoise by multiplying the average meter reading by the correct multiplication factor for the speed and the spindle. Obtain the correct factors using the factor finder slide rule provided with the viscometer.

The calculated viscosity should be within the individual site accepted tolerance of the certified value of the viscosity oil standard. If not, contact the manufacturer for repair instructions.

9. Remove and clean spindle prior to use for sample analysis.

(C) Sampling

Samples are normally provided by production.
Sample Analysis

1. Place the viscometer in "Standby Mode".

2. Allow the sample bottle to reach room temperature and then shake vigorously to re-mix any settled solids in the slurry. Check and record the temperature of the sample.

3. Fill a 600-mL, low-form Griffin beaker at least 3/4 full of slurry from the sample bottle.

   NOTE: Additional stirring may be necessary to eliminate any gross amounts of bubbles in the slurry after transfer to the beaker.

4. Select a #3 or #4 spindle assembly as follows:

   - For RPS VANTAGE® measurements, use #4 spindle.
   - For all other grades, use #3 spindle.

5. Insert the appropriate spindle into the beaker along the side of the beaker and move it to the center to remove any bubbles which may be trapped beneath the spindle.

6. Move the beaker position beneath the viscometer and attach the spindle so that the spindle remains submerged in the sample and is submerged to the groove on the spindle shaft.

7. Measure viscosity as follows:

   - Depress and hold the clutch (located on the back side of the meter), turn the motor switch "ON" (located on the right side of the meter), and set the motor speed selector to maximize the display reading for the expected viscosity. Following this procedure of having the clutch depressed at this point will prevent unnecessary wear.

   - Release the clutch and allow the dial to rotate until the pointer stabilizes at a fixed position on the dial, usually 8-10 revolutions.

   NOTE: At 100 rpm, this will take 10-15 seconds.

   - Depress the clutch lever with a thumb and turn the motor
"OFF" with the other hand, then read the position of the red pointer.

NOTE: If the pointer is not in view when the dial has come to rest, turn the motor switch "ON" and "OFF" rapidly until the pointer is visible in the window of the vision plate.

CAUTION: Always release the clutch while the spindle is still immersed, so that the pointer will float rather than snap back to "Zero".

- Take at least three readings, or more, if necessary, to obtain consistent readings. The readings should not have more than a 3% spread. If the spread is greater than 3%, more readings must be taken until 3 are recorded with no more than a 3% spread.

- Take an average of the three readings and record the value.

- Repeat the sequence to make additional measurements.

(E) Calculations

1. Calculate viscosity in centipoise by multiplying the average reading by the correct multiplication factor for the speed and the spindle. Obtain the correct factors using the factor finder slide rule provided with the viscometer.

   At 10 rpm, multiply the value by 100. At 100 rpm multiply the value by 10.

2. Report viscosity in centipoise, the rpm, and the temperature of the sample at measurement in the degrees centigrade.

IX. Quality Control

1. Recalibrate the viscometer, at 25 ± 1.0°C, at a frequency to be determined by each individual site, using a certified viscosity oil. The viscosity (oil) standards have an unlimited shelf life if kept uncontaminated. They should be changed at a frequency to be determined by each individual site. On an annual basis is recommended.
2. The degree of accuracy is maintained by frequent standardization of the viscometer against National Institute of Standards & Technology oils or against certified standards obtained from Brookfield. The results should be within the individual site accepted tolerance of the certified value, otherwise the instrument needs to be repaired.

X. Comments

The vendor recommends leaving the digital viscometer power switch "ON" between tests to minimize drifting of the viscometer display; however, the motor should be switched "OFF" when changing the spindle or changing samples.

XI. References

MSDS No. 2816CR.

XII. Appendix

None

The information set forth herein is furnished free of charge and is based on technical data that DuPont believes to be reliable. It is intended for use by persons having technical skill and at their own discretion and risk. Since conditions of use are outside our control, we make no warranties, express or implied, and assume no liability in connection with any use of this information. Nothing herein is to be taken as a license to operate under or a recommendation to infringe any patents.