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TITANIUM DIOXIDE

DETERMINATION OF SLURRY RHEOLOGY

METHOD: T4400.308.01.WP

TITANIUM DIOXIDE
(Ti-Pure®)

Rheology of Titanium Dioxide Slurries

I. Principle

Rheology is a measurement of the fluid flow characteristics of a liquid or slurry based on the relationship between flow rate (shear rate) and the force (shear stress) required to generate that flow rate.

A Hercules Hi-Shear Viscometer uses concentric cylinders to measure the resistance of a fluid to flow. Fluid is placed in the gap between the inner cylinder (called the "bob") and the outer cylinder (the sample cup of the instrument). The bob is rotated, generating a velocity gradient across the gap between the bob and the cup wall. The gradient is termed "Shear Rate" and is defined as the change in linear velocity between two fluid elements divided by their distance in centimeters. For normal testing, an "A" bob is used, which gives a gap of 0.05 cm.

The viscous resistance of the slurry sample to the spinning bob induces rotational deflection of the sample cup. A set of springs is used to measure the torque produced by this rotational deflection. For normal testing, a 50,000 dyne cm/cm spring is used.

The actual test consists of operating the bob from 0 to 1100 rpm and back to 0 rpm while the spring deflection is traced on a graph of rpm versus spring deflection. "Rheology" is reported as the spring deflection in centimeters that occurs at 500 rpm bob speed. As reported, "Rheology" is a simple relative value, useful for comparing a slurry sample against a standard or other samples.

II. Applicability

1. This method can be used to measure the rheology of titanium dioxide slurries.
2. "Rheology" can be converted to viscosity and rpm can be converted to shear rate in reciprocal seconds by the use of

appropriate equations or factors.

III. Limitations

Stable homogenous samples are required for accurate measurements. However, slurries tend to settle and agitating the sample is required for good mixing. This tends to introduce air bubbles which also interferes with accuracy. Care must be taken to get homogeneous samples.

IV. Sensitivity, Precision and Accuracy

A. Sensitivity

No data is available on sensitivity.

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 the method were as follows:

	Rheology in cm of deflection		
	\bar{X}	s	95% CL
TiO ₂ Slurry	1.85	0.05	± 0.13

The above data were calculated from eight replicate analyses of one sample performed by one technician over a period of one day.

2. Multiple Operator

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

	\bar{X}	s	95% CL
TiO ₂ Slurry	1.63	0.06	± 0.12

The above data were calculated from 32 replicate analyses of one sample performed by 4 technicians over a period of two weeks.

C. Accuracy

No data are available but results have been satisfactory based on experience.

V. Special Apparatus (Equivalent apparatus may be substituted)

1. Hercules Hi-Shear Viscometer, Model #24-4AC (or Model # ET-24-6), Kaltec Scientific Instruments Inc., 327E Ransom Street, Kalamazoo, MI. Standard equipment includes a 100,000 dyne cm/cm calibrated and matched spring set and a cup and five bobs.
2. A set of 50,000 dyne cm/cm spring set from same supplier. (At least 2 spare sets of springs should be kept in reserve).
3. Graph paper-Kueffel & Esser, 10x10 to the centimeter, 18 x 25 cm. A master is prepared and additional copies produced by xeroxing the original. (Do not make a copy from another copy as this tends to change the scale).

(Charts may be purchased from Kaltec. This would eliminate the possibility of having a "stretched" chart and would assure the proper chart was used each time).

VI. Reagents (Reagent grade except as noted)

1. Standard Test Fluid, Kaltec Scientific Instruments Inc., 327E Ransom Street, Kalamazoo, MI 49007.
2. Standard Test Fluid (Oil), (Viscosity standard), NIST Traceable or Certified from Brookfield. Brookfield Engineering Labs, Inc., 240 Cushing Street, Stoughton, MA 02072.

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 and 500 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. For more information, check the MSDS.

B. Procedure Hazards

Follow safety procedures outlined in the manufacturers operating manual for the Hercules Viscometer and normal laboratory safety procedures.

VIII. Procedure

A. Operating Conditions

1. The Hercules Viscometer is set up with:
 - a. "A" bob.
 - b. 50,000 dyne cm/cm spring set.
 - c. Maximum speed set at 1100 rpm.

B. Calibration

1. Routine calibration is not necessary. Monthly checking with a standard supplied by Kaltec and/or Brookfield (traceable to NIST) is recommended.
2. This standard is for use with the 50,000 dyne cm/cm spring set, the "A" bob and 0-1100 rpm. The instrument should be checked with the standard at installation, when a new standard is received and when a new set of springs is installed.

C. Sampling

Samples are normally supplied by manufacturing.

D. Sample Analysis

1. Set up Hercules Viscometer as instructed in the manufacturer's operating manual. Follow the manufacturer's operating instructions. Install the 50,000 dyne-cm/cm springs.
2. Put the cup in the holder and attach the "A" bob to the spindle of the Viscometer using moderate hand pressure. (The "bob" has a left hand thread).
3. Insert graph paper on the recording drum and check cord loops on the cup holder.
4. Pour approximately 41 mL of the sample into the viscometer cup.
5. Start viscometer, lower the bob to a position where the slurry level is slightly above the shoulder of the bob with only a slight excess of slurry on top of the bob. If a large excess is present, a false torque will result. In this case, remove the bob from the cup, clean the cup and bob, and start again with a new portion of slurry.
6. Adjust the pen to the zero position on the chart.
7. Press "auto" switch. The bob will start to rotate and increase speed uniformly until it reaches 1100 rpm and then return at the same rate to 0 rpm. Simultaneously the drum holding the chart paper will rotate and the pen will move up the chart and to the right. (The upward motion is controlled by the drive mechanism, but the motion to the right is controlled by the rheology of the fluid). When the pen reaches the top of the chart (1100 rpm bob speed), the pen reverses direction and returns gradually to zero. Push the "off" switch. The pen does not necessarily follow the same line on the down cycle as on the up cycle. (Some models are manual and are hand cranked. See manufacturers' operating manual).
8. Raise the bob from the cup, discard the sample and clean the equipment.

9. Remove the chart from the drum and record the horizontal deflection (average of the up and down trace) in cm to the nearest 0.1 cm at 500 rpm. On some curves there will be a horizontal deflection (yield stress) as the instrument begins.

E. Calculations

Rheology is calculated as follows:

$$\text{Rheology (deflection in cms)} = D$$

where:

$$D = \text{Deflection at 500 rpm (ave. of up \& down)}$$

IX. Quality Control

1. Check calibration of the Viscometer, on a frequency to be determined by each site, with NIST certified rheology oil.

X. References

Operating and Calibration Instructions from the manufacturer of the Viscometer.

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