WI _____ 210114.02 _____

T _____ 697 _____

BALLOT NO. ___ 02 SARG _____

DRAFT NO. ___ 02 _____

DATE _____ October 22, 2024 _____

WORKING GROUP
CHAIR _____ Gregg Reed _____

SUBJECT
CATEGORY ____ Coating & Graphic Arts _____

RELATED
METHODS ____ See "Additional Information"

Accelerated test for viscosity stability of clay slurries
(Proposed WITHDRAWAL of Classical Method T 697 cm-12)
(Re-balloted due to low percentage of voter turnout)
(Editorial changes shown)

1. Scope

1.1   This method describes a procedure for predicting the relative storage life of aqueous kaolin clay dispersions by measuring viscosity increase during storage at an elevated temperature.

1.2   Viscosity is measured by a low-shear rotating spindle apparatus as described in Appendix A of TAPPI T 648 "Viscosity of Coating Clay Slurry."

1.3   This procedure does not describe methods for determining dispersant requirements of clays, nor does it describe dry clay slurrying procedures. It describes only the testing procedures for a given clay slurry.

1.4   This procedure does not describe a method for determining the viscosity stability of other pigment slurries or for coating colors; however, this method may be useful for such systems.

2. Summary

A sample of clay-water slurry is stored at 60°C (140°F), and low-shear viscosity is measured as a function of storage time. From the increase of viscosity with time, the storage life of the slurry is estimated.

---

3.   Significance

3.1   Slurries of kaolin clay pigments in water are commonly used raw materials for paper manufacture. To obtain the high-solids slurries (67-70%) needed for economical shipment, yet maintain the low viscosity needed for easy handling, chemical deflocculants (commonly called dispersants) are added. These chemical deflocculants in general tend to decompose with time so that they lose their effectiveness in maintaining the low initial viscosity of the clay slurry. Unless a low viscosity can be maintained for a reasonable length of time, the slurry will thicken or "gel" so that it cannot be pumped by the normal slurry-handling equipment.

3.2   Clay slurries at high solids exhibit non-Newtonian flow behavior, i.e., their viscosity is a function of the shear rate applied. A given clay slurry may exhibit shear-thinning behavior at low-shear rates (thixotropy) yet show shear-thickening at high-shear rates (dilatancy). For a complete description of the flow behavior of a slurry, both low- and high-shear viscosities should be determined. The selection of a low-shear viscosity measurement for the present test is somewhat arbitrary, and it must be realized that it does not give a complete rheological description of the system. It has been found, however, that the low-shear viscosity measurement described in this test does correlate reasonably well with actual field observations as to the behavior of a slurry on pumping and mixing. If the low-shear viscosity of a slurry is high, it cannot readily be pumped even though its high-shear viscosity may be low.
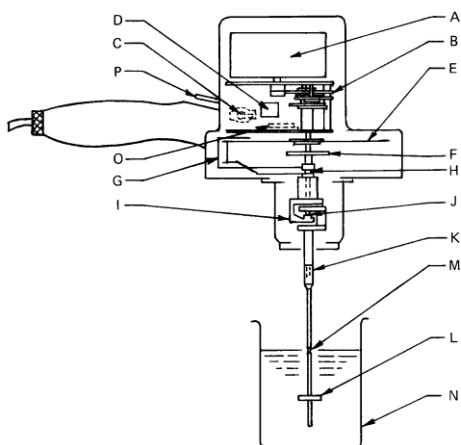

Fig. 1. Rotating spindle viscometer. A = synchronous motor used to power the viscometer; B = speed transmission driven by the motor; C = on-off switch for the motor; D = speed control knob; E = instrument dial driven by the output shaft of the speed transmission; F = beryllium copper spring, one end of which is attached directly to the viscometer dial; G = pointer; H = pivot shaft, upon which is mounted the collet, holding both the pointer G and spring; I = jewel support arm connected directly to the dial and to the gear transmission to the motor; J = pivot point mounted on pivot shaft; K = 3-56 left hand thread on the lower

point of the pivot shaft; L = spindle; M = immersion mark on shaft of each spindle; N = container for the specimen; O = bubble level; P = clutch lever. More detail can be found in the Appendix of TAPPI T 648 "Viscosity of Coating Clay Slurry."

4.   Apparatus

4.1   *Viscometer*, rotating spindle viscometer with a full-scale spring torque of 0.7187×10^3 N•m operating at 20 rpm with an appropriate spindle for mid-scale readings. A typical unit is shown in Fig. 1 and described in Appendix A of T 648. The newer digital versions may be used as well.

4.2   *Drying oven*, laboratory drying oven capable of being maintained at $105 \pm 3°C$.

4.3   *Oven or water bath*, for storing slurry at $60 \pm 1°C$.

4.4   *Containers*, glass containers of approximately 500-mL capacity with tightly fitting lids. (Ordinary 1-pint jars used for home canning are satisfactory.)

5.   Calibration

Calibrate the instrument with the standard-viscosity oil, using the sample spindle, volume, temperature, and container employed for the sample. Draw a calibration curve for the instrument based on at least two standard oils having higher and lower viscosities than the sample being measured. If the measured viscosity of the standard oil with an established factor differs by less than 2% from that given for the standard oil, the viscosity of the specimen may be found by applying a proportionate correction factor. If the difference is more than 2%, correct the instrument before use.

6.   Sampling

The sampling procedure or makeup procedure for the slurry is outside the scope of this method. It is assumed that the slurry sample is available for testing at an appropriate solids content. (The mixing procedure outlined in T 648 may be used for dry clays if desired.)

7.   Procedure

7.1   Determine the solids of the slurry by oven drying in accordance with TAPPI T 671 "Free Moisture in Fillers and Pigments (withdrawn)."

7.2   Place the slurry into the container to be used for storage at 60°C.

7.3   Adjust temperature of slurry to $25 \pm 1°C$.

7.4   Measure the low-shear viscosity on the viscometer at 20 rpm using the spindle that gives a scale reading on the viscometer between 20% and 80% of scale. Immerse the spindle to the mark in the mixture; start the viscometer rotating at 20 rpm. Place the spindle at an angle to reduce air entrapment under the blade. Allow the dial to rotate for 30 s, arrest it, and take the torque reading. Convert the torque reading to viscosity in mPa•s (cP) using the chart provided with the instrument.

7.5   Seal container tightly and weigh to nearest gram. Record weight.

7.6   Place container into storage at $60 \pm 1°C$.

7.7   After storing the slurry at 60°C for a known time, adjust temperature to $25 \pm 1°C$ and weigh to nearest gram. If this value is not within $\pm$ 2 g of that determined in 7.5, the solids have changed significantly and should be readjusted, or, preferably, the test should be repeated with a fresh sample.

7.8   Open the container and stir by hand with a spatula for 2 min.

7.9   Measure viscosity in accordance with 7.4.

7.10   Repeat steps 7.5 through 7.9 until desired aging time has elapsed or until slurry is no longer sufficiently fluid to measure.

8.   Report

8.1   Report the following items:

8.1.1   Type of sample, including dispersant, mixing conditions, and percent solids.

8.1.2   Viscosity in mPa • s (cP) at each measurement time interval.

8.1.3   The time in days that is required for the viscosity to increase to twice the initial viscosity as measured in 7.4. (This is conveniently determined by plotting the observed viscosity against storage time and reading the appropriate time.)

8.1.4   Alternatively, the actual viscosity measurements at indicated time intervals may be reported.

8.1.5   pH and the % solids at which it is measured.

9.   Precision

9.1   Precision, repeatability, and reproducibility are defined in TAPPI T 1200 "Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility."

9.2   The results in this section refer to the time measured according to 8.1.3.

9.2.1   Repeatability (within a laboratory) = 31%.

9.2.2   Reproducibility (between laboratories) = 34%.

9.3   The results in this section refer to the actual viscosity measurements after aging for 7 days under the prescribed conditions:

9.3.1   Repeatability (within a laboratory) = 7%.

9.3.2   Reproducibility (between laboratories) = 10%.

10.   Keywords

Viscosity, Stability, Clay, Slurry, Kaolin

11.   Additional information

11.1   Effective date of issue: To be assigned.

11.2   It is usually important to measure the pH of the slurry while viscosity measurements are made since storage stability is frequently strongly dependent on pH. The pH may be measured at the same solids or lowered to 20% solids in accordance with TAPPI T 667 "pH of Filler and Pigment Slurries (withdrawn)." pH normally decreases during aging, either because of dispersant decomposition or bacterial action.

11.3   This method may be applicable to other mineral pigment slurries or to coating color formulations, but reproducibility and repeatability have not been established for systems other than kaolin clay-water suspensions.

11.4   Convenient times for measuring viscosity are 1, 2, 4, 7, and 14 days, etc.

11.5   Coarser clays may settle to some extent during the test period to form a hard sediment at the bottom of the container. This will lower the effective solids for the slurry unless it is resuspended by stirring.

11.6   This method was reclassified in 1990 as a Classical Method by the Test Methods Management Committee of the TAPPI Board of Directors. In 1998, it was withdrawn due to lack of action in its regular review. It was reinstated in 2001.

11.7   Changes in this 2012 edition include references to newer digital equipment and more recommendations about the spindle in section 7.4.

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department.   ∎

Deleted: at the same time that