Designation: D3926 – 05 (Reapproved 2011)

Standard Test Methods for Percent Solids in Titanium Dioxide Slurries[1]

This standard is issued under the fixed designation D3926; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the determination of the weight percent of solids in aqueous slurries of titanium dioxide pigments by either the use of a gravity-convection oven (Method A), infrared radiation moisture analyzer (Method B), or a microwave drying system (Method C).

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This test standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

3. Summary of Test Method

3.1 *Oven Method:*

3.1.1 Slurry is weighed by difference into a tared aluminum foil dish, dried at 105°C in an oven for 1 h, cooled in a desiccator, and weighed.

3.2 *Infrared Method:*

3.2.1 Slurry is dried under infrared lamps for a specified time and temperature. The results are obtained directly form the unit's display panel.

3.3 *Microwave Method:*

3.3.1 2 to 4 g of slurry is placed between two glass fiber pads and dried in a microwave drying system. The results are obtained directly form the unit's display panel.

4. Significance and Use

4.1 These test methods are intended as a quick and reliable procedure for measuring the titanium dioxide pigment content of aqueous slurries. Included with the pigment content in the percent solids are the various nonvolatile additives used in preparing a stable slurry. Because the aluminum and silica oxide treatments on the more highly treated titanium dioxide pigments may change somewhat with prolonged drying, in the oven method the solids of the slurry are considered dry after heating at 105°C for 60 to 65 min. The high temperature associated with the infrared moisture analyzer may also effect a change in the aluminum and silica oxide treatment on highly treated TiO_2 products. Therefore, care in selection of time and temperature are critical to obtain accurate results with the infrared method. With the short duration of test associated with the microwave drying system, overdrying is not a concern.

5. Apparatus

5.1 *Oven Method:*

5.1.1 *Oven*—Laboratory oven capable of maintaining a temperature of 105 ± 2°C (Note 1). The oven may be a gravity-convection type or an oven with a low velocity, forced draft. An oven with a high-velocity, forced-draft air change, commonly used for baking finishes, is not suitable.

NOTE 1—The temperature in the oven must be constantly monitored. Many older ovens will no longer maintain ±2°C; some will maintain this tolerance for a while but occasionally the thermostat will "stick" and the temperature will vary considerably.

5.1.2 *Balance*—Laboratory analytical balance, accurate to 0.1 mg, with 1-g optical readout range for fast weighing.

NOTE 2—Periodically check the accuracy of the 1-g optical scale of the balance by use of a known 1-g weight; adjust the balance if needed. The zero adjustment of the optical scale needs to be checked at least every hour routinely and immediately if there is any possibility of a spill having occurred on the balance.

5.1.3 *Desiccator*—Standard laboratory desiccator utilizing an indicating drying medium.

5.2 *Infrared Method:*

5.2.1 *Infrared Moisture Analyzer*—Automated moisture analyzer combining infrared drying technology and a precision analytical balance in a single unit.

5.3 *Microwave Method:*

[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.31 on Pigment Specifications.

Current edition approved June 1, 2011. Published June 2011. Originally approved in 1980. last previous edition approved in 2005 as D3926 – 05. DOI: 10.1520/D3926-05R11.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

5.3.1 *Microwave Drying System*—A National Recognized Testing Laboratory (NRTL) approved 630 W microwave drying system with 50 g, 0.1 mg readability internal balance to measure moisture/solids from 0.1 to 99.99 %.

6. Reagents and Materials

6.1 *Oven Method:*

6.1.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D1193.

6.1.2 *Aluminum Foil Dish*—Disposable aluminum foil dishes, approximately 60-mm diameter by 18 mm high, 1 to 2 g in weight.

6.1.3 *Disposable Syringe or Dropper*—Disposable 2 or 3-mL syringes or 2 to 5-mL droppers.

6.2 *Infrared Moisture Analyzer:*

6.2.1 *Sample Holders* appropriate for the Infrared Moisture Analyzer.

6.2.2 *Disposable Syringe (2 or 3-mL) or Dropper.*

6.3 *Microwave Drying System.*

6.3.1 *Sample Holders* appropriate for the Microwave Drying System.

6.3.2 *Disposable Syringe (2 to 3-mL) or Dropper.*

7. Procedures

7.1 *Method A, Oven:*

7.1.1 Weigh two new, empty aluminum dishes each to 0.1 mg. For each dish, this is W_1. With a syringe add 2 mL of water to each dish.

7.1.2 Shake or stir the slurry sample until it is homogeneous and free of any settle material. If the container is transparent of translucent, the absence of settled material sticking to the bottom of the container can be ascertained visually. Otherwise insert a spatula or the like to make sure there is no settled material.

7.1.3 Immediately withdraw 0.4 to 0.8 g of slurry in a new, empty disposable syringe or dropper. The specified amount can be estimated by a prior trial in another syringe or dropper. Wipe off the slurry from the outside of the syringe or dropper with a clean, absorbent paper. Cover the sample bottle. Weigh the syringe or dropper and slurry to 0.1 mg. This is W_2.

7.1.4 Transfer the contents of the dropper into one of the weighed aluminum dishes. Add the slurry dropwise, gently shaking the dish to disperse the test specimen in the water. When no additional slurry can be transferred from the dropper, reweigh to 0.1 mg the dropper with any residual material inside. This is W_3.

7.1.5 Shake the sample of slurry briefly and withdraw a second 0.4 to 0.8 g of slurry in another new dropper. Weigh and transfer contents as in 7.1.3 and 7.1.4 to the second weighed aluminum dish.

7.1.6 Place the two dishes and contents directly onto the metal shelf in the oven at 105 ± 2°C for 60 to 65 min. Do not dry in the oven longer than the specified 65 min.

7.1.7 Remove the dishes from the oven, cool in the desiccator for 10 to 60 min, remove one dish at a time, and weigh immediately to the nearest 0.1 mg. This is W_4 for each dish. Do not cool longer than 60 min since there is a slight possibility of an equilibrium moisture exchange between the drying medium and some dried titanium dioxide pigments.

7.2 *Method B, Infrared Moisture Analyzer:*

7.2.1 Follow the manufacturer's recommendations for installation, set up, and calibration of the analyzer.

7.2.2 Determine a time and temperature schedule that produces slurry solids equal to those obtained with Method A, gravity convection oven. Program that schedule into the analyzer as recommended by the manufacturer.

7.2.3 Using a new, empty disposable syringe or dropper, transfer the required amount of sample from a TiO_2 slurry prepared as in 7.1.2 to a sample holder appropriate for the analyzer being used.

7.2.4 Follow the manufacturer's directions for drying the sample. When the end point is reached, the display will show the percent solids.

7.2.5 Repeat the procedure with a fresh sample of slurry.

7.3 *Method C, Microwave Drying System:*

7.3.1 Turn microwave unit on and follow manufacturer's directions on warm-up time if required before running the test.

7.3.2 Open unit door and place a pair of glass fiber pads on the balance pan.

7.3.3 Close the door and tare the panels following the manufacturer's instructions.

7.3.4 Prepare the slurry sample as in 7.1.2.

7.3.5 Open the oven door, remove the sample pads and use a new, empty disposable syringe or dropper to deposit 3 to 4 g of sample onto the rough side of one of the glass fiber pads (3 to 4 g of sample will create a bull's-eye the size of a quarter). Cover the sample with the other glass fiber pad, making sure to put the rough sides of the two pads together, gently squeeze the 2 pads together, and place on the balance pan.

7.3.6 Close the microwave drying system door, wait 3 s, and press RUN.

7.3.7 When the end point is reached, the display will show the percent solids and the elapsed time of the test.

7.3.8 Repeat the procedure with a fresh slurry sample, new glass fiber pads, and new disposable syringe or dropper.

8. Calculation

8.1 *Method A, Oven:*

8.1.1 For each of the duplicate measurements calculate the percent solids to two decimal places as follows:

$$\% \text{ Solids} = \frac{(W_4 - W_1)100}{W_2 - W_3}$$

where:

W_1 = weight of empty aluminum dish, g,
W_2 = weight of dropper plus slurry, g,
W_3 = weight of dropper after discharging slurry into dish, and g,
W_4 = weight of dish and slurry after drying, g.

Example:

W_1 = 1.4431
W_2 = 2.0894
W_3 = 1.4905
W_4 = 1.8158

$$\% \text{ solids} = \frac{(1.8158 - 1.4431)100}{2.0894 - 1.4905} = 62.23$$

8.1.2 Calculate the mean value of the duplicate measurements to two decimal places.

8.2 *Method B, Infrared Moisture Analyzer:*

8.2.1 % Solids will appear on the display panel. Calculate the mean value of the duplicate measurements to two decimal places.

8.3 *Method C, Microwave Drying System:*

8.3.1 % Solids will appear on the display panel. Calculate the mean value of the duplicate measurements to two decimal places.

9. Report

9.1 Round the calculated (Method A) or display panel solids (Method B and C) mean value to the nearest 0.1 % and report as percent solids. This rounded mean value is considered to be one result.

Example: solids = 62.2 %

9.1.1 For Method B, record the time, temperature, slope, and standby temperature conditions.

9.1.2 For Method C, record the elapsed time of the test.

10. Precision

10.1 *Method A, Oven:*

10.1.1 On the basis of an interlaboratory test of this method in which 17 operators in 15 laboratories analyzed 8 materials with solids contents at different levels, the within-laboratory standard deviation was found to be 0.12 % and the between-laboratory standard deviation was found to be 0.23 %. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

10.1.1.1 Two results, each the mean of duplicate determinations, obtained by the same operator at different times should be considered suspect if they differ by more than 0.34 % absolute.

10.1.1.2 Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 0.65 % absolute.

10.2 *Method B, Infrared Moisture Analyzer:*

10.2.1 On the basis of an interlaboratory test of this method in which 7 operators in 3 laboratories analyzed 4 materials with solids contents at different levels, the within-laboratory standard deviation was found to be 0.12 % and the between-laboratory standard deviation was found to be 0.44 %. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

10.2.2 Two results, each the mean of duplicate determinations, obtained by the same operator at different times should be considered suspect if they differ by more than 0.35 % absolute.

10.2.3 Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 1.34 % absolute.

10.3 *Method C, Microwave Drying System:*

10.3.1 On the basis of an interlaboratory test of this method in which 6 operators in 6 laboratories analyzed 4 materials with solids contents at different levels, the within-laboratory standard deviation was found to be 0.047 % and the between-laboratory standard deviation was found to be 0.225 %. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

10.3.2 Two results, each the mean of duplicate determinations, obtained by the same operator at different times should be considered suspect if they differ by more than 0.14 % absolute.

10.3.3 Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 0.68 % absolute.

11. Keywords

11.1 nonvolatile content; slurries; solids in titanium pigment slurries; titanium dioxide slurries