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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Percent Solids in Titanium Dioxide Slurries[1]

This standard is issued under the fixed designation D 3926; 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 This test method covers the determination of the weight percent of solids in aqueous slurries of titanium dioxide pigments.

1.2 *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:*

D 1193 Specification for Reagent Water[2]

3. Summary of Test Method

3.1 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.

4. Significance and Use

4.1 This test method is 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 oxide modifiers on some titanium dioxide pigments may change somewhat with prolonged drying, in this method the solids of the slurry are considered dry after heating at 105°C for 60 to 65 min.

5. Apparatus

5.1 *Oven*—Laboratory oven capable of maintaining a temperature of 105 ± 2°C (Note 1). The oven may be a gravityconvection 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.

5.2 *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.

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

6. Reagents and Materials

6.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

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

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

7. Procedure

7.1 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.

7.2 Shake or stir the slurry sample until it is homogeneous and free of any settled material. If the container is transparent or 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.

7.3 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.

7.4 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.

7.5 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 6.3 and 6.4 to the second weighed aluminum dish.

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[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.31 on Pigment Specifications.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

7.6 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.

7.7 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.

8. Calculation

8.1 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 % solids $= \frac{(1.8158 - 1.4431)100}{2.0894 - 1.4905}$
W_4 = 1.8158 $= 62.23$

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

9. Report

9.1 Round the calculated 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 %

10. Precision

10.1 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 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.

10.1.2 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.

11. Keywords

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