

TITANIUM DIOXIDE IN PAINTS AND THERMOPLASTICS

(An Arizona Method)

1. SCOPE

- 1.1 This test method may be used to make a rapid estimation of the amount of Titanium Dioxide pigment in paints and thermoplastics, particularly those of white color used for traffic control markings.
- 1.2 The method is a non-instrumental, gravimetric method, which involves removing, successively, all accompanying constituents of the material, leaving only the Titanium Dioxide analyte. The isolated analyte is weighed directly.
- 1.3 The method is applicable for paints and for thermoplastics, with or without glass beads.
- 1.4 **The procedure in this method consists of two parts. Part A is for paints; Part B is for thermoplastics.**
- 1.5 This test method involves hazardous material, operations, or equipment. This test method does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine the applicability of any regulatory limitations prior to use.
- 1.6 See Appendix A1 of the Materials Testing Manual for information regarding the procedure to be used for rounding numbers to the required degree of accuracy.

2. APPARATUS

- 2.1 Requirements for the frequency of equipment calibration and verification are found in Appendix A3 of the Materials Testing Manual. Apparatus for this test procedure shall consist of the following:
 - 2.1.1 600 mL beaker, graduated.

- 2.1.2 Whatman No. 40 ashless filter paper, 125 mm, or equivalent.
- 2.1.3 A balance or scale capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M 231, except that the readability and sensitivity of any balance or scale utilized shall be at least 0.0001 gram.
- 2.1.4 50 mL porcelain crucible.
- 2.1.5 Filtration funnel, 7.5 cm, 60°, long stem.
- 2.1.6 Watch glass, 3 inch.
- 2.1.7 Glass stirring rod.
- 2.1.8 Muffle furnace, capable of maintaining a constant temperature of 450 °C.
- 2.1.9 Drying oven, capable of maintaining a constant temperature of 110 °C.
- 2.1.10 Dessicator.
- 2.1.11 Hot plate.

3. REAGENTS

- 3.1 Concentrated Hydrochloric Acid Solution.
- 3.2 Concentrated Nitric Acid Solution.
- 3.3 Demineralized water.

4. PROCEDURE

PART A (FOR PAINTS):

- 4.1.1 Weigh 5.0000 ± 0.2500 grams of paint into a crucible. Record the weight of the paint specimen as "B" to the nearest 0.0001 gram.
- 4.1.2 Place crucible and specimen into the drying oven at temperature of 110 °C.

- 4.1.3 After 2 hours, remove crucible, place into cold muffle furnace, and set the temperature to 450 °C.
- 4.1.4 Ignite specimen at 450 °C for 1 hour after the temperature of 450 °C is attained.
- 4.1.5 Remove crucible with ignition residue and allow to cool to room temperature.
- 4.1.6 Place crucible with residue into 300 mL beaker.
- 4.1.7 Cautiously, dropwise, add 30 mL Concentrated Hydrochloric Acid to crucible.
- 4.1.8 Cautiously add 10 mL Concentrated Nitric Acid to crucible.
- 4.1.9 After digestion of the residue is complete, add 250 mL of demineralized water to beaker, add 1 boiling chip, cover with watchglass, and place on the hot plate.
- 4.1.10 Heat beaker contents to boiling and boil for 15 minutes.
- 4.1.11 Remove beaker from hotplate and filter contents, carefully washing and decanting titanium dioxide, leaving boiling chip and glass beads, if any, in the beaker. Wash thoroughly with demineralized water.
- 4.1.12 Tightly fold filter and place into tared crucible. Record crucible weight as "A," to the nearest 0.0001 gram.
- 4.1.13 Place crucible and contents into cold furnace and set temperature to 450 °C.
- 4.1.14 Ignite specimen at 450 °C for 1 hour after temperature of 450 °C is attained.
- 4.1.15 Remove crucible and allow to cool to room temperature in dessicator.
- 4.1.16 Weigh crucible and contents. Record weight as "C," to the nearest 0.0001 gram.

4.2 **PART B (FOR THERMOPLASTICS):**

4.2.1 Weigh 5.0000 ± 0.2500 grams of thermoplastic into a crucible. Record the weight of the thermoplastic specimen as "B" to the nearest 0.0001 gram.

4.2.2 Place crucible and specimen into cold muffle furnace and set the temperature to 450 °C.

4.2.3 Proceed as specified in Subsections 4.1.4 through 4.1.16.

5. **CALCULATIONS AND REPORT**

5.1 Calculate Titanium Dioxide, in percent, using the following formula:

$$\text{Titanium Dioxide, \%} = \frac{C - A}{B} \times 100$$

5.2 Report Titanium Dioxide to the nearest 0.001%.