This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: D4451 – 22

Standard Test Method for Pigment Content of Paints by Low-Temperature Ashing[1]

This standard is issued under the fixed designation D4451; 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 pigment content of paints and several traffic marking materials (thermoplastic and preformed tape) by low-temperature furnace ashing. Some organic pigments may be lost by this method and some water or moisture contained in pigments will be lost.

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 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3723 Test Method for Pigment Content of Water-Emulsion Paints by Low-Temperature Ashing

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

3. Summary of Test Method

3.1 The specimen is transferred to a tared porcelain dish, dried (if necessary) at 105 °C, and heated on a burner. The dish and specimen are transferred to a muffle furnace and heated at 450 °C. The dish and specimen are reweighed and the pigment (ash) content calculated.

4. Apparatus

4.1 *Muffle Furnace,* maintained at 450 °C ± 25 °C.

4.2 *Circulating Oven,* maintained at 105 °C ± 2 °C.

4.3 *Porcelain Dishes,* 90 mm diameter.

4.4 *Plastic Disposable Syringe,* 10 mL capacity.

4.5 *Burner,* Meker type.

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4]

5.2 *Toluene.*

6. Procedure

6.1 *Liquid-Paint:*

6.1.1 Mix the sample until homogeneous, preferably on a mechanical shaker. If air bubbles become entrapped in the paint, stir it by hand.

6.1.2 Draw slightly more than 10 g of the paint under test into a 10 mL syringe and weigh to 0.1 mg. Weigh a porcelain dish with a paper clip for use as a stirrer, and record the mass. Transfer about 5 mL of toluene to the dish. Add 10 g of the test material into the toluene. Reweigh the syringe to 0.1 mg and calculate the specimen weight. Mix well using the paper clip. Place the dish in the oven at 105 °C for 30 min.

6.1.3 Remove and heat at the lowest temperature possible over a Meker burner in a fume hood until the material ignites. Do not leave the dish on the burner after the flame has subsided. Transfer to the muffle furnace and proceed as in 6.4.

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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[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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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6.2 *Preformed Traffic Marking Tape*—Cut about a 10 g square of the product. Remove the adhesive by pulling it off or by using an appropriate solvent. Save the beads that are knocked off by this process and weigh with the tape. Dry the specimen for 30 min at 105 °C to remove the solvent. Cool in a desiccator. Weigh an empty porcelain dish and record its mass. Transfer the specimen along with the loose beads into the tared porcelain dish and weigh to 0.1 mg. Heat at the lowest temperature possible over a Meker burner in a fume hood until the material ignites. Do not leave the dish on the burner after the flame has subsided. Transfer to the muffle furnace and proceed as in 6.4.

6.3 *Thermoplastic Traffic Marking Material*—This material may be delivered in block or powdered form. Transfer about 400 g of the sample to a quart container and heat in a forced draft oven at 400 °F until completely melted (may take as long as 4 h). Stir vigorously until well mixed. Pour about 10 g of the sample into an aluminum dish and let it cool. Remove the 10 g wafer from the aluminum dish and weigh to 0.1 mg. Weigh an empty porcelain dish and record its mass. Transfer the wafer to the tared porcelain dish. Heat the dish at the lowest temperature of a Meker burner in a fume hood until the specimen ignites. Do not leave the dish on the burner after the flame has subsided. Transfer to a muffle furnace and proceed as in 6.4.

6.4 Place the porcelain dish in a muffle furnace in a fume hood at 450 °C (see Test Method D3723). Heat overnight or until no further carbonaceous material is noted. Cool in a desiccator and reweigh the dish plus the residue to 0.1 mg. Calculate the residue as percent pigment or, in the case of thermoplastic material and preformed tape, as pigment and beads.

7. Calculation

7.1 Calculate the percent pigment, P, as follows:

$$P = (C - A)/S \times 100 \quad (1)$$

where:

C = weight of dish and specimen after ignition in furnace, g,
A = weight of dish alone, g, and
S = specimen weight used, g.

8. Precision

8.1 The precision estimates are based on an interlaboratory study in which one operator in 7 different laboratories analyzed in duplicate on two days six samples of commercial whole paint or thermoplastic material containing 50 to 80 % pigment. The results were analyzed statistically in accordance with Practice E180 and the within-laboratory coefficient of variation was found to be 0.10 % relative at 36 df and the between-laboratories coefficient of variation 0.25 % relative at 30 df. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

8.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 0.28 % relative.

8.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 0.72 % relative.

9. Bias

9.1 Bias cannot be determined because there are no standards for pigment content of paints.

10. Keywords

10.1 ignition; low temperature ashing; pigment; pigment content of paints

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