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**Designation: D 4451 – 02**

## Standard Test Method for Pigment Content of Paints by Low-Temperature Ashing<sup>1</sup>

This standard is issued under the fixed designation D 4451; 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.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-1, 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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### 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 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 3723 Test Method for Pigment Content of Water-Emulsion Paints by Low-Temperature Ashing<sup>2</sup>

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals<sup>3</sup>

### 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.<sup>4</sup>

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.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 06.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 15.05.

<sup>4</sup> *Reagent Chemicals, American Chemical Society Specifications*, 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.

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. Transfer about 5 mL toluene to a porcelain dish tared with a paper clip for use as a stirrer. Add 10 g of the material into the toluene. Reweigh the syringe to 0.1 mg and calculate the specimen weight. Mix well on a magnetic stirrer. 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. Do not leave the dish on the burner after the flame has subsided. Transfer to the muffle furnace and proceed as in 6.4.

6.2 *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 the specimen along with the loose beads to 0.1 mg into a tared porcelain dish. Heat in a fume hood at the lowest temperature of a meker burner in a furnace hood until the material catches fire. 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. Transfer to a tared porcelain dish. Heat the dish at the lowest temperature of a meker burner in a fume hood until the specimen catches fire. 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 D 3723). 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 E 180 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

9.1 ignition; pigment;

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

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