Designation: D 3723 – 84 (Reapproved 1999)

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Pigment Content of Water-Emulsion Paints by Low-Temperature Ashing[1]

This standard is issued under the fixed designation D 3723; 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 a procedure for the pigment content determination of water-based paints. It is applicable only to pigments that do not decompose or lose weight at temperatures below 500°C. Such pigments include most metal oxides, silicates, and a majority of anhydrous inorganic salts.

1.2 Many water-based paints contain pigments and organic colorants that lose water of hydration or decompose at this temperature. The residual ash should be carefully inspected for changes in color or texture that could indicate a pigment alteration and hence lead to erroneous results. Caution should therefore be exercised when applying this test method to samples containing unknown pigment compositions.

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 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. Significance and Use

3.1 This test method is used by paint producers and consumers for product process control and for product acceptance.

4. Apparatus

4.1 *Oven*, forced draft, maintained at 105 ± 2°C.

4.2 *Furnace*, muffle, maintained at 450 ± 25°C.

4.3 *Syringe*, 5-mL.

4.4 *Aluminum Foil Dish*, 58 mm in diameter by 18 mm high with a flat bottom. The bottom of the dish should be as nearly flat as possible so that a uniform film is produced.

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.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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

5.3 *Ammonium Hydroxide*[4]—Add 1 volume of concentrated NH_4OH (sp gr 0.90) to 3 volumes of water.

6. Procedure

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

6.2 Draw approximately 1.5 g of the test paint into a 5-mL syringe and weigh to 1 mg. Add the paint dropwise (about 30 drops) into a tared-aluminum foil dish that contains 2 mL of water (5.2). Swirl the dish during the addition and continue the swirling until the specimen is completely dispersed. Reweigh the syringe to 1 mg. Transfer between 0.4 and 0.6 g of sample to the dish. If not, adjust the transferred volume and prepare a new specimen. If the specimen agglomerates or forms a lump that cannot be dispersed, a drop or two of ammonia (5.3) may facilitate the dispersement. If the lumping persists, discard the specimen and prepare a new one. Prepare a duplicate specimen in the same manner.

6.3 Dry the specimen and dishes in the 105 ± 2°C oven for a minimum of 1 h after making certain that the dishes are level. If the coating does not uniformly cover the bottom of a dish, prepare a new specimen and repeat.

NOTE 1—The percent nonvolatile content, *N*, at 105°C may be determined by drying dishes to constant weight and calculating as follows:

$$N = \frac{A - B}{S} \times 100 \quad (1)$$

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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 D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved Oct. 26, 1984. Published January 1985. Originally published as D 3723 – 78. Last previous edition D 3723 – 78.

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

[3] *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.

[4] For guidance in the safe handling of ammonium hydroxide consult the Manufacturing Chemists Association's Chemical Safety Data Sheet.

where:
A = weight of dish and specimen after heating,
B = weight of dish alone, and
S = specimen weight.

6.4 Transfer the dishes to a muffle furnace and heat at 450 ± 25°C for 1 h. Remove from the furnace, cool in a desiccator and weigh.

7. Calculation

7.1 Calculate the percent pigment content, P, as follows:

$$P = \frac{C - B}{S} \times 100 \quad (2)$$

where:
C = weight of the dish and specimen after ignition in the furnace,
B = weight of the dish alone, and
S = specimen weight.

8. Precision and Bias [5]

8.1 *Precision: Pigment Content*—On the basis of an interlaboratory test of this test method in which eighteen operators in nine laboratories analyzed three materials containing three pigment levels between 24 and 28 %, the within-laboratory standard deviation was found to be 0.24 % absolute and the between-laboratories 0.38 % absolute. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

8.1.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 0.66 % absolute.

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 1.05 % absolute.

8.2 *Bias: Pigment Content*—On the basis of the same study, the mean of two determinations should be within 5 % relative of the formula pigment content of a waterborne paint containing only inorganic pigments.

8.3 *Precision: Nonvolatile Content*— On the basis of an interlaboratory test of this test method in which fourteen operators in seven laboratories analyzed three materials containing three nonvolatile levels between 43 and 48 %, the within-laboratory standard deviation was found to be 0.18 % absolute and the between-laboratories standard deviation was found to be 0.445 % absolute. Based on these standard deviations the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

8.3.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 0.49 % absolute.

8.3.2 *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 1.23 % absolute.

9. Keywords

9.1 low-temperature ashing; pigment content; water emulsion paints

[5] Supporting data are available from ASTM Headquarters. Request RR:D01.1014.