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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Nonvolatile and Pigment Content of Electrocoat Baths[1]

This standard is issued under the fixed designation D 5145; 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 These test methods cover the characterization of electrocoat baths through the determination of nonvolatile content of inorganic pigment content.

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 1193 Specifications for Reagent Grade Water[2]

D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings[3]

E 180 Practice for Determining the Precision Data of ASTM Methods for Analysis and Testing of Industrial Chemicals[4]

3. Summary of Test Method

3.1 Two specimens are accurately weighed into aluminum weighing dishes. The dishes are placed in an oven at 110°C for 1 h, reweighed to obtain the nonvolatile matter content and, if required, placed in a muffle furnace at 500°C for 2 h and weighed a third time to obtain the inorganic pigment content.

4. Significance and Use

4.1 The nonvolatile content and pigment content are measures of total solids and inorganic pigment solids, respectively, in electrocoat paints. In addition to production quality control, these properties are important in maintaining electrocoat baths in the optimum range.

4.2 Other test methods for determining nonvolatile content of paint and paint related materials are described in Method D 2832.

5. Apparatus

5.1 *Analytical Balance* with a sensitivity of 0.1 mg.

5.2 *Aluminum Weighing Dishes*, 57 mm in diameter and 17 mm deep. These commercial dishes may contain a lubricant used during their manufacture. This should be removed by heating the aluminum dishes on a hot plate at 300°C until vapors are no longer visible. Store the dishes in a desiccator until needed.

5.3 *Syringes*, 5-mL, disposable variety.

5.4 *Oven* circulating, maintained at 110 ± 2°C.

5.5 *Muffle Furnace*, maintained at 500 ± 15°C.

6. Reagents

6.1 *Purity of Water*—References to water shall be understood to mean water conforming to Type II of Specification D 1193.

7. Sampling and Sample Preparation

7.1 Obtain the sample while the electrocoat bath is under proper circulation so a uniform sample is obtained. In the case of a ultrafiltrate sample, the material should be thoroughly mixed or stirred prior to drawing the sample, thereby ensuring uniformity.

7.2 After sampling, prior to removing the test specimen, it is mandatory the sample be shaken or stirred until it is homogeneous and free of any settled material. This is particularly important if there is a delay between sampling the bath and performing this test procedure. The absence of settled material should be ascertained visually or by inserting a spatula and scraping the bottom of the container. Continue to shake or stir the sample until specimens are taken for measurement. *This Point is Very Important.*

NONVOLATILE CONTENT

8. Procedure

8.1 Weigh two aluminum dishes separately, each to 0.1 mg and record as W_1.

8.2 Using a syringe, withdraw 1.0 to 1.5 of the well mixed sample, then quickly weigh the syringe to 0.1 mg, recording this weight as W_2. Transfer the entire contents of the syringe into the aluminum dish. Reweigh the empty syringe to 0.1 mg and record as W_3. In the case of ultrafiltrate clear liquids or of low solids paints, increase the specimen size to 5 mL and preheat at 60°C for 2 h. Duplicate this step with the second aluminum dish (8.1).

8.3 Add a few millilitres of water to the specimen in the

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

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 15.05.

aluminum dishes prior to placing them in the oven. This facilitates uniform spreading of the material. Place the dishes in the 110°C oven for 1 h.

8.4 Remove the dishes from the oven and allow to cool to room temperature in a dessicator. Reweigh them to 0.1 mg and record the weights as W_4.

8.5 Retain the dishes for measurement of inorganic pigment content as detailed in a following section of these test methods.

9. Calculation

9.1 Calculate the percent nonvolatile content as follows:

$$\% NV = \frac{W_4 - W_1}{W_2 - W_3} \times 100 \quad (1)$$

where:
W_1 = weight of empty aluminum dish, g,
W_2 = weight of syringe filled with sample, g,
W_3 = weight of empty syringe, g, and
W_4 = weight of dish and contents after 1 h at 110°C, g.

10. Precision and Bias

10.1 Precision is based on an interlaboratory study in which the operators in each of ten laboratories analyzed in duplicate on 2 days, four different electrocoat-bath samples with nonvolatile contents ranging from 0.30 to 25.2 % The results were analyzed statistically in accordance with Practice E 180. The interlaboratory coefficient of variance was 1.6 % at 30 df and the interlaboratory coefficient of variation was 2.3 % at 27 df. Based on the these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

10.1.1 *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 4.6 % relative.

10.1.2 *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 6.7 % relative.

10.2 No bias has been determined for this test method.

INORGANIC PIGMENT CONTENT

11. Procedure

11.1 Place the weighing dishes used for the nonvolatile content determination in the muffle furnace at 500°C for 2 h.

11.2 After 2 h, remove the dishes from the muffle furnace, transfer them to a dessicator and allow to cool to room temperature. After cooling, remove dishes and weigh to 0.1 mg, recording these weights at W_5.

NOTE 1—If an organic char is still present in the aluminum dish after 2 h in the muffle furnace, continue heating in the furnace until no char is present.

12. Calculation

12.1 Calculate the percent inorganic pigment as follows:

$$\% \text{ Inorganic Pigment (EC Bath)} = \frac{W_5 - W_1}{W_2 - W_3} \times 100 \quad (2)$$

where:
W_1 = weight of empty aluminum dish, g,
W_2 = weight of syringe filled with sample, g,
W_3 = weight of empty syringe, g, and
W_4 = weight of aluminum dish and contents after heating in muffle furnace, g.

13. Precision and Bias

13.1 Precision estimates are based on an interlaboratory study in which the operators in ten different laboratories analyzed in duplicate on 2 days, four electrocoat bath materials containing inorganic pigment content ranging from 0.17 to 4.52 weight %. The results were analyzed statistically in accordance with Practice E 180. The intralaboratory coefficient of variation was 2.2 % relative at 40 df and the interlaboratory coefficient of variation was 10.8 % at 36 df. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

13.1.1 *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 6.3 % relative.

13.1.2 *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 31.1 % relative.

13.2 No bias has been determined for this test method.

14. Keywords

14.1 electrocoat bath; muffle furnace; nonvolatile content; pigment content; ultrafiltrates