



Standard Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments¹

This standard is issued under the fixed designation D 280; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover procedures for determining hygroscopic moisture (and other matter volatile under the test conditions) in pigments.

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.*

METHOD A—FOR PIGMENTS THAT DO NOT DECOMPOSE AT 110°C

2. Apparatus

2.1 *Weighing Bottle*, wide-mouth, cylindrical, glass (flat form, about 30 mm in height and about 70 mm in diameter), provided with a ground-in glass stopper. Or, an aluminum moisture dish (about 90 mm in diameter and about 50 mm in depth) with a tightly fitting cover.

2.2 *Oven* in which a temperature of from $110 \pm 2^\circ\text{C}$ is maintained.

2.3 *Analytical Balance*.

3. Procedure

3.1 Weigh accurately the glass weighing bottle and stopper or the aluminum moisture dish with cover. Place a specimen of from 3 to 5 g of the pigment in the clean, dry weighing bottle or in the clean, dry aluminum moisture dish. Insert the stopper (or cover) and weigh to 0.1 mg. Subtract the weight of the vessel from the total weight to obtain the weight of sample used in the test. Remove the stopper (or cover) and place it and the bottle (or dish) containing the specimen in an oven that has been previously heated to $110 \pm 2^\circ\text{C}$, heat for 2 h at a temperature of 105 to 110°C . Replace the stopper (or cover), cool in a desiccator, and weigh. Calculate the total loss in weight as percent of moisture and other volatile matter.

METHOD B—FOR PIGMENTS THAT DECOMPOSE AT 110°C

4. Apparatus

4.1 *Weighing Bottle*, glass, as described in 2.1.

4.2 *Open-Tube Manometer* made of glass tubing 6 mm in diameter, filled with mercury to approximately 860 mm, fitted with rubber pressure tubing attached to a T-joint leading to the desiccator and the pump. A suitable low-pressure gage may be used in place of the manometer.

NOTE 1—The difference in levels of the mercury in the manometer when the system is in operation, subtracted from the barometer reading taken at the same time, gives the pressure of the system in millimetres of mercury.

4.3 *Desiccator*, glass, having a hole at the side or in the cover, constructed with heavy walls to withstand a vacuum of one atmosphere. The hole at the side shall be closed with a one-hole rubber stopper carrying a glass tube with a rubber tube connection and a pinchcock or with a glass stopcock ground to fit the tubulature.

4.4 *Oil Vacuum Pump*,² able to achieve and hold a vacuum of 3 mm.

4.5 *Analytical Balance*.

5. Procedure

5.1 Weigh accurately the glass weighing bottle and stopper. Place a specimen of from 1 to 3 g of the pigment in the clean, dry weighing bottle, insert the stopper and weigh to 0.1 mg. Subtract the weight of the vessel from the total weight to obtain the weight of sample used in the test.

5.2 Remove the stopper and place it and the bottle containing the specimen in the desiccator containing *fresh*, anhydrous magnesium perchlorate.³ Close the desiccator, attach to the pump, and *gradually* evacuate until the pressure is constant at 3 mm or less (Note 2). Close the pinchcock or stopcock, stop the pump, and let stand at room temperature (21 to 32°C) for 24 h.

¹ These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.31 on Pigment Specifications.

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² The “Hyvac” oil pump was used in these test methods. This is the sole source of supply of this pump known to the committee at this time. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

³ Available from Chemical Suppliers under the name Anhydrous or Dehydrite.

5.3 Slowly admit air that has been dried by passage through *fresh* magnesium perchlorate to the desiccator by means of the pinchcock or stopcock, remove the cover, quickly replace the stopper in the weighing bottle, and weigh. Repeat the evacuations and weighings until the loss in weight does not exceed 0.5 mg in 24 h. Calculate the total loss in weight as percent of moisture and other volatile matter (Note 3).

NOTE 2—Caution should be used in evacuating glass desiccators. The vacuum should be applied gradually. Desiccators will maintain a vacuum for a greater length of time when a thin film of a suitable stopcock

lubricant is applied to the ground surfaces. Desiccators when evacuated should be wrapped in towels or covered by other means to prevent possible injury to the operator.

NOTE 3—This is an empirical method and the details should be strictly followed. The determination of the true hygroscopic moisture content of very finely divided pigments is very difficult, if not impossible in some cases.

6. Keywords

6.1 moisture; pigment; volatile

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