



# Standard Test Methods for Common Properties of Certain Pigments<sup>1</sup>

This standard is issued under the fixed designation D1208; 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 certain properties of pigments. The procedures appear in the following order:

Dry Pigments	Sections
Loss on Ignition and Ash	4
Matter Soluble in Water	5
Hydrogen Ion Concentration (pH Value)	6
Alkalinity or Acidity by Titration	7 and 8
Water Content (Distillation Method)	9 and 10
Pigment Pastes in Oil	
Water Content (Distillation Method)	9 and 10
Pigment Content of Paste in Oil	11
Total Volatile Matter in Paste in Oil	12

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

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

[D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation](#)

[D280 Test Methods for Hygroscopic Moisture \(and Other Matter Volatile Under the Test Conditions\) in Pigments](#)

[D1135 Test Methods for Chemical Analysis of Blue Pigments](#)

<sup>1</sup> 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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<sup>2</sup> 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.

## E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

## 3. Significance and Use

3.1 This collection of test methods is used by pigment producers and paint manufacturers for process control, for product acceptance, and for research and development.

### LOSS ON IGNITION AND ASH

## 4. Procedure

4.1 Dry about 5 g of the sample at 105 ± 2°C for 2 h. Transfer about 1 g of the dried pigment, weighed to 0.1 mg, to a previously ignited, weighed porcelain crucible, and ignite at 900 to 1000°C for 20 min. Cool in a desiccator and weigh. Heat again for 10 min at 900 to 1000°C to check the loss in weight.

4.2 *Calculation*—Calculate the percent of loss on ignition, *L*, and of ash, *A*, as follows:

$$L = \frac{L_w}{S_1} \times 100 \quad (1)$$

$$\left( A = \frac{W_a}{S_1} \times 100 \right)$$

where:

*L<sub>w</sub>* = loss in weight on ignition, g,

*A* = ash, %,

*W<sub>a</sub>* = weight of ash, g, and

*S<sub>1</sub>* = specimen weight, g.

### MATTER SOLUBLE IN WATER

## 5. Procedure (Note 1)

5.1 Weigh about 10 g of the sample to 1 mg, and place in a 400-mL beaker. Add 100 mL of water (Note 2), boil for 5 min cool, and transfer quantitatively to a 250-mL volumetric flask. Dilute with water to 250 mL, mix, and allow to settle. Filter the supernatant liquid through a dry paper (Note 3) and discard the first 25 mL. Evaporate 100 mL of the clear filtrate to dryness in a weighed flatbottom dish, preferably in an oven at 105 ± 2°C. Cool and weigh.

NOTE 1—This test method is not suitable for use with iron blue pigment. Use the conductivity method given in Test Methods D1135.

NOTE 2—If the pigment is found to be strongly water-repellent, wet the sample with a small amount of alcohol or carry out a preliminary washing with chloroform.

NOTE 3—Water-dispersible pigments must be filtered with a suitable filter pad or the method may not be applicable.

5.2 *Calculation*—Calculate the percent of matter soluble in water,  $M$ , as follows:

$$M = \frac{R_1 \times 2.5}{S_2} \times 100 \quad (2)$$

where:

$R_1$  = residue weight, g, and

$S_2$  = specimen weight, g.

## HYDROGEN ION CONCENTRATION (pH VALUE)

### 6. Procedure

6.1 Determine hydrogen ion concentration in accordance with either of the following methods:

6.1.1 *Electrometric Method (Preferred Procedure)*—Weigh about 5 g of the sample to 10 mg, place in a 150-mL beaker, and add 50 g of warm distilled water, freshly boiled to remove carbon dioxide. Mix well by means of a glass rod and cool to 25°C. Measure the pH of the mixture in accordance with Test Method E70.

6.1.2 *Colorimetric Method*—Weigh about 10 g of the sample to the nearest 10 mg, place in a 250-mL glass-stoppered Erlenmeyer flask, and add 150 g of warm distilled water, freshly boiled to remove carbon dioxide. Stopper the flask and shake vigorously for about 1 min. Cool to 25°C. Let stand 1 h to allow the pigment to settle. Decant the clear liquid and determine its pH at once by means of a suitable indicator, using a block comparator or similar colorimetric method.

NOTE 4—A block comparator or equivalent device is necessary in order to compensate for the color of the extract.

6.2 *Report*—In reporting test results, state the particular method used.

## ALKALINITY OR ACIDITY BY TITRATION

### 7. Reagents

7.1 *Methyl Orange Indicator Solution*—Dissolve 1 g of methyl orange in 1 L of reagent water.

7.2 *Sodium Hydroxide, Standard Solution* (0.02 N).

7.3 *Sulfuric Acid, Standard Solution* (0.02 N).

### 8. Procedure

8.1 Shake 20 g of the dry pigment with 200 mL of distilled water for 5 min, let settle, and filter through a dry filter paper into a dry beaker, discarding the first 10 mL of the filtrate. Transfer 100 mL of the clear filtrate to a flask, add 3 drops of methyl orange indicator solution, and titrate to the end point with 0.02 N sulfuric acid ( $H_2SO_4$ ), if alkaline, or 0.02 N sodium hydroxide (NaOH) solution, if acid.

8.2 *Calculation*—Calculate the acidity or alkalinity, expressed as milligrams of NaOH equivalent to 1 g of the sample, as follows (1 mL of 0.020 N NaOH solution is equivalent to 0.8 mg of NaOH):

$$\text{Alkalinity or acidity} = \text{mL of NaOH or } H_2SO_4 \times 0.8 \quad (3)$$

## WATER CONTENT

### 9. Apparatus

9.1 *Distillation Apparatus*—A water distillation apparatus consisting of a 250-mL roundbottom flask, a straight-tube reflux condenser, and a graduated receiving trap similar to Fig. 1 of Test Method D95.

9.2 *Heat Source* consisting of a gas burner and oil bath, or an electric heater of the enclosed element type.

### 10. Procedure (Note 5 and Note 6)

10.1 Place 50 g of the sample in the flask and add 100 mL of toluene or of petroleum solvent having a boiling point between 110 and 120°C. Mix well. Add more solvent, if necessary, to cover the pigment.

10.2 Rinse the inner tube of the condenser with a little toluene or petroleum solvent just before starting the distillation, so as to wet the inner surface completely. Distill at a moderate rate until the volume of condensed water no longer increases (about 3 h). If any water is lodged in the condenser tube at the end of the test, wash it down with solvent or with a brush wetted with solvent.

10.3 *Calculation*—Calculate the weight percent of water,  $W$ , as follows (assuming that 1 mL of water weighs 1 g):

$$W = \frac{W_c}{50} \times 100 \quad (4)$$

where  $W_c$  = water collected, mL.

NOTE 5—This test method is not suitable for pigments that release combined water under the conditions of the test.

NOTE 6—Test methods especially adapted for the determination of moisture in iron blue pigments are given in Test Methods D1135.

NOTE 7—Test Methods designed for the determination of free water (as opposed to water of crystallization) in dry pigments are given in Test Methods D280.

## PIGMENT CONTENT OF PASTE IN OIL

### 11. Procedure

11.1 Select a portion of the well-mixed sample that is free of skins. Weigh to 10 mg, about 15 g of the sample in a weighed 60-mL centrifuge tube. Add about 30 mL of petroleum ether, mix thoroughly with a glass rod, and wash the rod with more petroleum ether (Note 8). Centrifuge at a moderate speed until well settled. Decant the clear supernatant liquid, and repeat the extraction twice with 40 mL of petroleum ether and once with 40 mL of ethyl ether. After decanting the ether, set the tube on top of a warm oven for 10 min and then in an oven at  $105 \pm 2^\circ C$  for 2 h. Cool and weigh.

NOTE 8—Other solvents or solvent mixtures may be used, provided that they leave no residue on evaporation.

11.2 *Calculation*—Calculate the percent pigment content,  $P$ , as follows:

$$P = \frac{R_2}{S_3} \times 100 \quad (5)$$

where:

$R_2$  = residue weight in centrifuge tube, g and

$S_3$  = specimen weight, g.

$$V = \frac{L}{S_4} \times 100 \quad (6)$$

where:

$L$  = loss in weight on heating, g, and

$S_4$  = specimen weight, g.

## TOTAL VOLATILE MATTER IN PASTE IN OIL

### 12. Procedure

12.1 Weigh to 1 mg a flatbottom dish about 80 mm in diameter containing a piece of stiff wire 100 mm long. Leave the wire in the dish throughout the test. Place about 4 g of the sample, weighed to 1 mg in the dish. Spread the specimen over the bottom by means of the wire and heat for 2½ h in an oven at  $105 \pm 2^\circ\text{C}$ . If a skin forms, break it up at intervals during the heating by means of the wire. Cool the dish and contents in a desiccator and weigh. Heat again for 30 min to check the loss in weight.

12.2 *Calculation*—Calculate the percent volatile matter in the sample,  $V$ , as follows:

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## PRECISION AND BIAS

### 13. Precision and Bias

13.1 Precision data are not available at this time. When they are available the appropriate statements will be added.

13.2 *Bias*—No bias has been determined for this test method.

### 14. Keywords

14.1 analytical; ash; common products; loss on ignition; pigments; volatile water; water content; water solubility ph