



Standard Test Methods for Common Properties of Certain Pigments¹

This standard is issued under the fixed designation D 1208; 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 *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 95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation²
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³
- D 1135 Test Methods for Chemical Analysis of Blue Pigments³
- E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁴

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_w = loss in weight on ignition, g,

A = ash, %,

W_a = weight of ash, g, and

S_1 = 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 D 1135.

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:

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.

Current edition approved Nov. 10, 1996. Published January 1997. Originally published as D 1208 – 52 T. Last previous edition D 1208 – 84 (1989)^{ε1}.

² *Annual Book of ASTM Standards*, Vol 05.01.

³ *Annual Book of ASTM Standards*, Vol 06.03.

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

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

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 E 70.

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 D 95.

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 D 1135.

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 D 280.

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 ± 2°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.

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 ±2°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:

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

where:

L = loss in weight on heating, g, and

S_4 = specimen weight, g.

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

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).