



Standard Test Methods for Para Red and Toluidine Red Pigments¹

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

1. Scope

1.1 These test methods cover para red and toluidine red pigments (toners) in the dry form commercially known as “pure.”

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 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller³
- D 1193 Specification for Reagent Water⁴
- D 3335 Test Method for Low Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy³
- D 3624 Test Method for Low Concentrations of Mercury in Paint by Atomic Absorption Spectroscopy³

3. Significance and Use

3.1 These test methods are used to determine the purity and some physical properties of para red and toluidine red pigments. This information is significant to pigment producers and to coatings manufacturers.

4. Reagents

4.1 *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.⁵ 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.

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

4.3 *Chloroform*.

4.4 *Hydrochloric Acid (1+1)*—Mix equal volumes of concentrated hydrochloric acid (HCl, sp gr 1.19) and water.

4.5 *Nitric Acid (3+5)*—Mix 3 volumes of concentrated nitric acid (HNO₃, sp gr 1.42) with 5 volumes of water.

4.6 *Potassium Hydroxide, Alcoholic Solution*—Dissolve 10 g of potassium hydroxide (KOH) in 90 mL of ethyl alcohol (95 %).

4.7 *Sodium Hydroxide (100 g/L)*—Dissolve 100 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

5. Solubility in Chloroform

5.1 Place about 0.05 g of the dry pigment in a 50-mL Nessler tube, add 40 mL of chloroform, and warm slightly, stirring with a glass rod. Compare this solution with a similarly prepared solution of a standard sample of known purity. Complete solubility of the pigment in chloroform is indicated if a clear orange red solution is obtained.

NOTE 1—Any turbidity indicating an impurity may be separated conveniently by extracting 5 g of the pigment in a soxhlet extractor, using chloroform as the solvent. The residue may then be examined and estimated.

6. Qualitative Test for Identity and Purity

6.1 To 10 mL of the chloroform solution (5.1) add 2 mL of alcoholic potassium hydroxide (KOH) solution. An intense violet coloration indicates the presence of para red toner, (1-(*p*-nitro-phenylazo)-2-naphthol). A wine red coloration indicates the presence of toluidine red toner (1-(2-nitro-*p*-tolylazo)-2-naphthol). It is well to make side-by-side comparisons, using a standard pigment as control.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 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 *Annual Book of ASTM Standards*, Vol 06.03.

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

⁴ *Annual Book of ASTM Standards*, Vol 11.01.

⁵ *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 Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

NOTE 2—Infrared spectrophotometry may also be employed for this purpose if equipment is available and suitable reference spectra have been agreed upon by purchaser and seller.

7. Ash

7.1 Transfer about 2 g of the sample (weighed to 0.1 mg) to a tared porcelain or platinum crucible and heat at red heat until all organic matter is destroyed. Cool and weigh.

NOTE 3—For examination for low concentrations of lead and mercury, refer to Test Methods D 3335 and D 3624.

7.2 Calculate the percent of ash present as follows:

$$\text{Ash, \%} = (R/S) \times 100$$

where:

R = residue, g, and

S = sample used, g.

8. Resistance to Acids and Alkalis

8.1 Place about 0.5 g of the dry pigment in a 50-mL beaker and add about 40 mL of HNO₃ (3+5). Stir well and allow the pigment to settle. Decant the liquid and filter it. The filtrate should show no more color than a reference pigment mutually agreed upon by the purchaser and the seller. Repeat using HCl

(1+1). Repeat using NaOH solution (100 g/L), omitting the filtration.

9. Moisture and Other Volatile Matter

9.1 Determine the percent moisture and other volatile matter in accordance with Test Methods D 280.

10. Coarse Particles

10.1 Determine the percent of coarse particles in accordance with Test Methods D 185.

11. Mass Color and Character of Tint

11.1 Determine the mass color and character of tint in accordance with Test Method D 387.

12. Precision and Bias

12.1 Data are not available to determine the precision and bias of these test methods. There are no plans at present to obtain such data. The methods have been in use for several years and are considered acceptable.

13. Keywords

13.1 para red pigment; analysis of; pigment; toluidine red pigment; analysis of; toner; para red and toluidine red

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