

Designation: D 1686 – 96 (Reapproved 2000)

Standard Test Method for Color of Solid Aromatic Hydrocarbons and Related Materials in the Molten State (Platinum-Cobalt Scale)¹

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1. Scope

1.1 This test method covers the visual measurement of the color of thermally stable solids melting below 150°C. It is applicable only to materials in which the color-producing bodies present have light absorption characteristics quite similar to those of the standards used.

1.2 The following applies to all specified limits in this test method: for purposes of determining conformance with this test method, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E 29.

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. For specific hazard statements see Sections 7 and 9.

2. Referenced Documents

2.1 ASTM Standards:

- D 1193 Specification for Reagent Water²
- D 3438 Practice for Sampling and Handling Naphthalene, Maleic Anhydride, and Phthalic Anhydride³
- D 3852 Practice for Sampling and Handling Phenol and Cresylic Acid³
- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications⁴

2.2 Other Document:

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200⁵

³ Annual Book of ASTM Standards, Vol 06.04.

3. Significance and Use

3.1 Color by this test method is a measure of colorproducing impurities present in the thermally stable solids. This test method is suitable for setting specifications and for use as an internal quality control tool.

4. Apparatus

4.1 *Color Comparison Tubes*—Matched 100-mL, tall-form Nessler tubes, provided with ground-on, optically clear, glass caps. Tubes should be selected so that the height of the 100-mL graduation mark is 300 ± 3 mm above the bottom of the tube. The use of heat-resistant tubes is preferred for safety reasons.

4.2 Color Comparator—A color comparator constructed to permit visual comparison of light transmitted through tallform, 100-mL Nessler tubes in the direction of their longitudinal axes. The comparator should be constructed so that white light is reflected off a white plate and directed with equal intensity through the tubes, and should be shielded so that no light enters the tubes from the side.

4.3 *Oven*—An oven, preferably of the forced draft type and capable of maintaining a constant temperature $\pm 1^{\circ}$ C in the range up to 150°C. Alternatively, the use of an aluminum heating block provided with proper temperature control or other similar equipment is permissible.

5. Reagents

5.1 *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.⁶ 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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type IV reagent water

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² Annual Book of ASTM Standards, Vol 11.01.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

⁶ 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 Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

conforming to Specification D 1193.

5.3 *Cobalt Chloride* (CoCl₂·6H₂O).

5.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

5.5 *Potassium Chloroplatinate* (K₂PtCl₆).

6. Standards

6.1 *Platinum-Cobalt Stock Solution*— Dissolve 1.245 g of K₂PtCl₆ and 1.000 g of CoCl $_2$ ·6H₂O in water. Add 100 mL of HCl and dilute to 1 L with water. This solution has a color of 500.⁷

TABLE 1 Platinum-Cobalt Color Standards^A

Color Standard No.	Stock Solution, mL	Color Standard No.	Stock Solution, mL
5	1	35	7
10	2	40	8
15	3	50	10
20	4	60	12
25	5	70	14
30	6	100	20

^A Other color standards may be prepared by proportional dilution.

6.2 *Platinum-Cobalt Standards*⁸—From the stock solution, prepare color standards, as given in Table 1, by diluting the required volumes to 100 mL with water in the Nessler tubes. If desired, these standards may be made permanent by sealing on the caps with a suitable colorless cement.

7. Hazards

7.1 Consult OSHA regulations, supplier's Material Safety Data Sheets, and local regulations for all materials used in this test method.

7.2 **Warning:** When handling molten solids in open tubes, adequate ventilation must be provided and proper protection should be used to prevent thermal burns.

8. Sampling

8.1 Sample the material in accordance with Practices D 3438 or D 3852.

9. Procedure

9.1 Melt approximately 150 g of the sample and simultaneously preheat a Nessler tube and cap in an oven maintained at constant temperature at 10 to 20° C above the solidification point of the sample. The sample must not be heated to more than 20° C above its solidification point.

9.2 As soon as the sample is completely liquid, mix by stirring with a clean dry glass rod, then quickly fill the preheated Nessler tube to the 100-mL mark with the sample and cap the tube. Place the tube in the comparator and immediately compare with the standards.

Note 1—A variation of more than 2 to 3 mm in depth can affect the test results.

9.3 When measuring the color of solids that sublime, some difficulty may be encountered due to condensation of solid on the cap. In such a case, the Nessler tube may be reheated in the oven after first loosening the cap, or, the color may be measured using an open Nessler tube without a cap (**Warning**, see 9.3.1). In this latter case the caps should also be removed from the standards.

9.3.1 **Warning:** Make sure to provide adequate ventilation if open tubes are used.

9.4 In no case shall this test method be applied if the molten sample contains any visible turbidity.

10. Report

10.1 Report as the color, the number of the standard that most nearly matches the sample. In the event that the color lies midway between two standards, report the darker of the two.

10.2 If, owing to small differences in hue between the sample and the standards, an exact match cannot be obtained, but an estimate is possible, report the range over which an approximate match is obtained, and report the sample as "off hue."

10.3 If, owing to large differences in hue between the sample and the standards, no estimate is possible, report the sample as "no match."

11. Precision and Bias

11.1 Intermediate Precision (formerly called Repeatability)—Results in the same laboratory should not be considered suspect unless they differ by more than the following amounts:

Platinum-Cobalt Color	Intermediate Precision
Under No. 50	2.5
No. 50 to No. 100	5
Over No. 100	10

11.2 *Reproducibility*—Results submitted by two laboratories should not be considered suspect unless they differ by more than the following amounts:

Platinum-Cobalt Color	Reproducibility	
Under No. 50	5	
No. 50 to No. 100	10	
Over No. 100	15	

11.3 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, bias has not been determined.

12. Keywords

12.1 color; cresylic acid; maleic anhydride; naphthalene; phenol; phthalic anhydride; platinum-cobalt; solid aromatic hydrocarbons

 $^{^7}$ The stock solution with color No. 500 may be purchased as such from chemical supply firms. Use of the purchased standard is satisfactory.

⁸ The preparation of these platinum-cobalt standards was originally described by A. Hazen, *American Chemical Journal*, Vol 14, p. 300 (1892). The description given in Test Method D 1686 is identical with that given in the "Standard Methods for the Examination of Water and Sewage," American Public Health Assn., Tenth Edition, 1955, p. 88. A description is also given by W. W. Scott, "Standard Methods of Chemical Analysis," D. Van Nostrand Co., Inc., Fifth Ed., Vol 2, p. 2048.

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