



Designation: D3366 – 18

Standard Test Method for Color of Maleic Anhydride in the Molten State and After Heating (Platinum-Cobalt Scale)¹

This standard is issued under the fixed designation D3366; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope*

1.1 This test method covers the determination of the visual measurement of the color of maleic anhydride melt before and after prolonged heating under specified conditions of time and temperature. Color values are expressed in terms of platinum-cobalt standards.

1.2 This test method covers the range 0 to 100 color standard numbers.

1.3 In determining the conformance of the test results using this method to applicable specifications, results shall be rounded off in accordance with the rounding-off method of Practice E29.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements see Sections 7, 10.2.1, and 10.4.2.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

¹ This test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee D16.02 on Oxygenated Aromatics.

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² 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.

D1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)

D1686 Test Method for Color of Solid Aromatic Hydrocarbons and Related Materials in the Molten State (Platinum-Cobalt Scale)

D3438 Practice for Sampling and Handling Naphthalene, Maleic Anhydride, and Phthalic Anhydride

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

D8005 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 Other Document:³

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200

3. Summary of Test Method

3.1 A specimen is melted at 60°C.

3.2 A freshly melted specimen is filled to mark into a Nessler tube and compared with Platinum-Cobalt color standards.

3.3 After heating for 2 h at 140°C, the specimen is again compared to the color standards.

4. Significance and Use

4.1 The color of maleic anhydride can be an indication of the purity of these materials. High colors normally indicate contamination or decomposition. This test method is suitable for setting specifications and for use as an internal quality control tool.

5. Apparatus

5.1 *Color Comparison Tubes*—Matched 100-mL, tall-form Nessler tubes, provided with ground-on, optically clear, glass

³ Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, http://www.access.gpo.gov.

*A Summary of Changes section appears at the end of this standard

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.

5.2 *Color Comparator*, constructed to permit visual comparison of light transmitted through tall-form, 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.

5.3 *Oven*—An oven, preferably of the forced draft type and capable of maintaining a constant temperature $\pm 1^\circ\text{C}$ in the range up to 140°C . Alternatively the use of an aluminum heating block provided with proper temperature control or other similar equipment is permissible.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all the reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ 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.

6.2 *Purity of Water*—References to water shall be understood to mean water, conforming to Type IV of Specification D1193.

6.3 *Cobalt Chloride*, ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$).

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

6.5 *Potassium Chloroplatinate*, (K_2PtCl_6).

7. Hazards

7.1 Consult current OSHA regulations, supplier's 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 and Handling

8.1 Sample the material in accordance with Practice D3438.

9. Calibration and Standardization

9.1 *Platinum-Cobalt Stock Solution*—Dissolve 1.245 g of K_2PtCl_6 and 1.000 g of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ in water. Add 100 mL of HCl and dilute to 1 L with water. This solution is defined as color standard No. 500. The absorbance of the 500 Platinum-

Cobalt Stock Solution in a cell having a 10-mm light path with distilled water in a matched cell as the reference solution must fall within the limits given in Table 1.

TABLE 1 Absorbance Tolerance Limits for No. 500 Platinum-Cobalt Stock Solution

Wavelength	Absorbance
430	0.110 to 0.120
455	0.130 to 0.145
480	0.105 to 0.120
510	0.055 to 0.065

9.2 *Platinum-Cobalt Standards*—From the stock solution prepare color standards in accordance with Table 2 by diluting the required volumes to 100 mL with water in volumetric flasks. When properly sealed and stored these standards are stable for at least one year. 500 Platinum-Cobalt standards may also be purchased.

10. Procedure

10.1 Melt approximately 150-g specimen and simultaneously preheat a Nessler tube and cap in an oven, electric heating block, or other similar equipment held at a temperature of 60°C for maleic anhydride.

10.2 As soon as the specimen 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 specimen and cap the tube. Place the tube in the comparator and immediately compare with the standards and record as the color of the specimen in the molten state.

10.2.1 Use caution and wear appropriate safety equipment when handling hot samples and equipment.

10.2.2 Report as the color, the number of the standard that most nearly matches the specimen. For samples between 20 and 100 Pt-Co color, estimate the color between the standards below and above the specimen.

10.3 Place the filled Nessler tube in an oven, electric heating block, or other similar equipment which has been regulated at 140°C ($\pm 1^\circ\text{C}$). Allow the tube to remain in the block for 2 h

TABLE 2 Platinum-Cobalt Color Standards

Color Standard Number	Stock Solution, mL	Color Standard Number	Stock Solution, mL
1	0.20	19	3.80
2	0.40	20	4.00
3	0.60	25	5.00
4	0.80	30	6.00
5	1.00	35	7.00
6	1.20	40	8.00
7	1.40	45	9.00
8	1.60	50	10.00
9	1.80	55	11.00
10	2.00	60	12.00
11	2.20	65	13.00
12	2.40	70	14.00
13	2.60	75	15.00
14	2.80	80	16.00
15	3.00	85	17.00
16	3.20	90	18.00
17	3.40	95	19.00
18	3.60	100	20.00

⁴ *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.