



Designation: D3366 – 95 (Reapproved 2003)^{e1}

Standard Test Method for Color of Maleic Anhydride and Phthalic 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 reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Editorial changes were made throughout in February 2003.

1. Scope

1.1 This test method covers the determination of the visual measurement of the color of maleic and phthalic anhydride melt before and after prolonged heating under specified conditions of time and temperature. Color values are expressed in terms of platinum-cobalt standards. This test method covers the range 0 to 100 color standard numbers.

1.2 The following applies to all specified limits in this standard: for purposes of determining conformance with this standard, an observed value or a calculated value shall be rounded of “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 E29.

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 Section 7.

2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

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

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

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

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

Current edition approved Feb. 10, 2003. Published April 2003. Originally approved in 1974. Last previous edition approved in 1995 as D3366 – 95. DOI: 10.1520/D3366-95R03E01.

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.

2.2 Other Document:

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200³

3. Summary of Test Method

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

3.2 After heating for 2 hours at a prescribed temperature, the specimen is again compared to the color standards.

4. Significance and Use

4.1 The color of maleic anhydride and phthalic anhydride can be an indication of the purity of these materials. High colors normally indicate contamination.

5. Apparatus

5.1 *Color Comparison Tubes*—Matched sets of 50-mL Nessler tubes having a total length of about 300 mm. The height of the 50-mL gradation mark shall be within 200 to 250 mm above the exterior bottom of the tube. In a given set the gradation marks shall not vary by more than 3 mm. The use of heat resistant glass is required.

5.2 *Color Comparator*, constructed to permit visual comparison of light transmitted through the 50-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.)

NOTE 1—For convenience of operation, an electrically heated, insulated comparator tube may be used to prevent the solidification of maleic or phthalic anhydride.

5.3 *Electric Heating Block*—An electrically heated aluminum block, such as shown in Fig. 1, having the following operating characteristics:

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

TABLE 1 Platinum—Cobalt Color Standards^A

Color Standard No.	Stock Solution mL	Color Standard No.	Stock Solution mL
5	0.5	35	3.5
10	1.0	40	4.0
15	1.5	50	5.0
20	2.0	60	6.0
25	2.5	70	7.0
30	3.0	100	10.0

^A Other color standards may be prepared by proportional dilution in steps of 20 as follows: No. 120, No. 140, No. 160, etc.

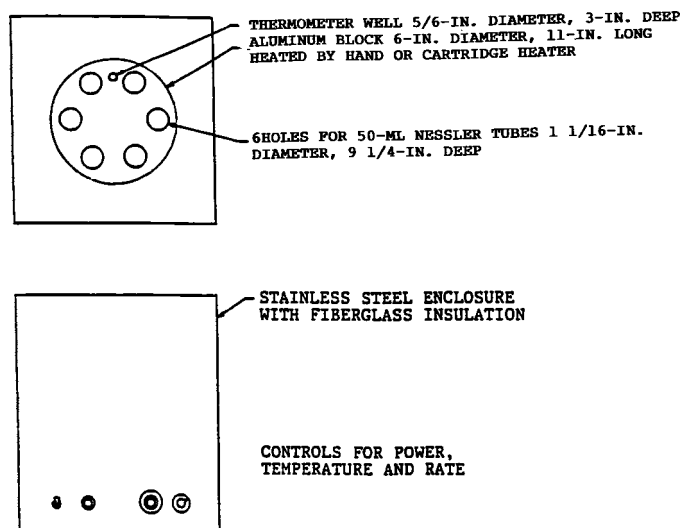


FIG. 1 Heat Stability Block

Maleic anhydride temperature regulation, °C	140 ± 2
Heating time for specimen from 60 to 140°C, minutes	25 ± 5
Phthalic anhydride temperature regulation, °C	250 ± 2
Heating time for specimen from 150 to 250°C, minutes	25 ± 5
Temperature gradient, specimen well to thermometer well, °C max	2

The operating characteristics of the heating block used are critical.

6. Reagents

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*—Unless otherwise indicated reference to water shall be understood to mean reagent water conforming to Specification D1193, Type II.

6.3 *Cobalt Chloride* (CoCl₂·6H₂O).

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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

6.4 *Hydrochloric Acid* (specific gravity 1.19)—Concentrated hydrochloric acid (HCl).

6.5 *Potassium Chloroplatinate*—(K₂PtCl₆).

7. Hazards

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

8. Sampling

8.1 Sample in accordance with Practice D3438.

9. Standards

9.1 *Platinum-Cobalt Stock Solution*⁵—Dissolve 1.245 g of K₂PtCl₆ and 1.000 g of CoCl₂·6H₂O in water. Add 100 mL of HCl and dilute to 1 L with water. This solution is defined as color standard No. 500.

9.2 *Platinum-Cobalt Standards*⁶—From the stock solution, prepare color standards by diluting the required volumes as given in Table 1, to 50 mL with water in the Nessler tubes. When not in use, protect these standards from contamination and evaporation by the use of suitable caps on the tubes.

10. Procedure

10.1 Melt an approximately 75-g specimen (Note 2) and simultaneously preheat a Nessler tube in an oven, electric heating block, or other similar equipment held at a temperature of 60°C for maleic anhydride and 150°C for phthalic 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 50-mL mark with the specimen and place in the comparator; immediately compare with the standards and record as the color of the specimen in the molten state.

10.3 Place the filled Nessler tube in the heating block, which has been regulated at the appropriate temperature:

For maleic anhydride	140 ± 2°C
For phthalic anhydride	250 ± 2°C

Use a small ring of glass wool around the top of the hole to support in a vertical position and to seal off the dead air space between the tube and the heating block. Allow the tube to remain in the block for 2 hours (which includes 90 min at the test temperature). Protect tubes adequately from contamination during the test period.

NOTE 2—Place a small quantity of glass wool in the bottom of each hole to ensure against breakage of Nessler tubes and thermometers.

⁵ The stock solution with color No. 500 may be purchased as such from chemical supply firms. Use of the purchased standards is satisfactory.

⁶ The preparation of these platinum-cobalt standards was originally described by Hazen, A. *American Chemical Society Journal*, Vol. 14, 1892, p. 300. The description given in this test method and in ASTM Test Method D1209, Test for Color of Clear Liquids (Platinum-Cobalt Scale), which appears in the *Annual Book of ASTM Standards*, Vol 06.03, is identical with that given in the *Standard Methods for the Examination of Water and Sewage*, American Public Health Association, Tenth Edition, 1955, p. 88. A description is also given by W. W. Scott in *Standard Methods of Chemical Analysis*, D. Van Nostrand Co., Inc., Sixth Edition, Vol. 2, Part B, p. 2424.